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PART I

A RECORDING THERMOBALANCE FOR MATERIALS RESEARCH

Part I - Constant Heating Rate Thermogravimetry To 1400°C

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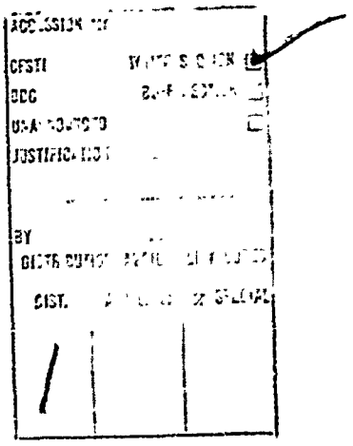

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PART I

A RECORDING THERMOBALANCE FOR MATERIALS RESEARCH

PART I - CONSTANT HEATING RATE THERMOGRAVIMETRY TO 1400°C.

N. J. OLSON

FOREWORD

This report was prepared by the University of Dayton Research Institute, Dayton, Ohio, under USAF Contract AF 33(615)-3961. This contract was initiated under Project No. 7340, "Nonmetallic and Composite Materials," Task No. 734001, "Thermally Protective Plastics." This work was administered under the direction of the Nonmetallic Materials Division, Air Force Materials Laboratory, with Mr. J. M. Kelble, MAN, acting as project engineer.

The contributions of Mr. R. A. Grant in glassware fabrication and Mr. R. W. Farmer in providing many candid technical criticisms are acknowledged with appreciation.

Many of the items compared in this report were commercial items that were not developed or manufactured to meet any Government specification, to withstand the tests to which they were subjected, or to operate as applied during this study. Any failure to meet the objectives of this study is no reflection on any of the commercial items discussed herein or on any manufacturer.

This report covers work conducted from March 1965 to March 1968. The manuscript was released by the author in April 1968 for publication.

This report has been reviewed and is approved.

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ABSTRACT

This report describes a means of modifying a recording thermobalance for routine constant heating rate thermogravimetry to 1400°C with an optional temperature capability of 1750°C. During operation, a sample is suspended beneath a linear variable differential transformer (LVDT) system by precision springs. Any sample weight change varies the relative position of the springs resulting in a proportional voltage being generated by the LVDT system. The conditioned voltage is then plotted on the Y^1 axis of an X, Y^1, Y^2 recorder. Temperature is recorded on the X carriage axis via a platinum, platinum+13% rhodium, platinum (ground) thermocouple placed into the sample powder. A synchronous time base generator supplies a time readout for the Y^2 axis. The constant heating rate is closely regulated by a spherical furnace/SCR controller/time base recorder system. The atmosphere of the suspension chamber is air-freed and helium-filled by room temperature evacuation/purge cycles before the run. A run typically involves a -325 sieve 80 mgm dried powder sample to 1400°C at 8.5°C/min in helium purging flow. Recorder ranges are typically 0-20 mg/5 inch, 0-1400°C/20 inch, and 0-200 min/5 inch.

In this thermobalance, the thermocouple wires are insulated by an alumina sheath which also serves as the crucible holder. Tungsten wire/mercury pool/tungsten rod electrical contacts result in minimum spring suspension effect. The thermocouple signal is filtered and grounded to shield against electromagnetic furnace noise normally found at high temperatures.

Typical thermograms are given for phenolic resin and polybenzimidazole/carbon cloth laminate samples. The latter material, which is of interest for hyperthermal ablative use, underwent pyrolysis beyond the typical analysis limit of 1000°C and approached a constant weight at 1400°C. The poor grinding qualities of reinforced plastics required a unique lathe machining method to produce a fine powder sample.

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I. INTRODUCTION

The recording thermobalance is a versatile and useful laboratory tool for materials research. Typically, the weight change of a sample causes mechanical movement of a spring. This movement is converted into an electrical signal. The signal is then recorded as a function of time when the sample is cooking at a fixed temperature. If the temperature is programmed at a constant rate of change, the second variable can be either temperature or time.

The successful aerospace use of plastics as coatings, structural members and particularly for ablative thermal protection has resulted in many thermogravimetric studies of these materials. Some of the new polyaromatic resins with good ablative performance and many refractory reinforcements continue to pyrolyze at temperatures higher than the typical 1000°C limit of commercial thermobalances. Therefore, there has been little work within this interesting temperature region. Thermogravimetry above 1000°C imposes severe operational and reliability requirements. Versatile quartz furnace tubes are no longer suitable and alumina or other refractories must be substituted for quartz or porcelain components. At these temperatures a thermocouple or other temperature sensor is ideally placed within or attached to the sample. The leadout wire cannot appreciably effect the sensitive weighing system and must be free from electromagnetic pickup due to furnace operation.

This report describes the modification of a commercial recording thermobalance, the Aminco Thermo-Grav Model 4-4430, for routine constant heating rate work to 1400°C with a 1750°C optional mode of operation. Figures 1 and 2 are photographs of the unit before and after modification. The main changes made to the original unit include: (a) a two-pen X, Y^1, Y^2 recorder with sample weight, sample temperature, and run time readout; (b) a 3-wire platinum, platinum+ 13% rhodium, platinum (ground) thermocouple for electromagnetic noise reduction and direct sample emplacement without disturbing the spring suspension system; (c) a furnace/controller/recorder system; (d) a synchronous time base generator/interruptive marker for a wide range of time spans; (e) micrometric adjusters for the suspension system and LVDT; and (f) a gas purge/vacuum system for controlled atmosphere runs. Additional new devices for further operational ease and reliability include an alumina thermocouple sheath/crucible holder, furnace tube and crucible, special glassware with tungsten electrode leadouts, a spring housing constant temperature bath and circulator.

Basic principles, new instrumentation and operational procedures for the thermobalance are described in considerable detail in view of the possible adoption of similar innovations by other interested workers. Typical experimental results are summarized for three reinforced plastics of interest for aerospace use.

This report also describes a means of reducing bulk samples of carbonaceous fiber reinforced materials to a fine powder by using a machine lathe.

II. THEORY OF OPERATION

The weighing mechanism is the key element of any balance. This thermobalance uses vertical springs that are rigidly held at one end by an adjustable fixture as shown on the functional diagram (Figure 3). The two precision springs are enclosed by a Pyrex housing of double-wall construction. A constant temperature water circulator controls the temperature of the inner annulus. This is necessary to protect the sen- -ve springs against ambient temperature changes. A reference spring housing temperature is visually monitored during a run using a chamber thermocouple and digital readout counter.

A Pyrex main support rod with an eyelet end is hooked to the springs. The rod supports a linear variable differential transformer (LVDT) core, calibration pan and dashpot-type dampener by means of Teflon bushings. A thermocouple sheath and crucible hang from the other end of the support rod.

A change in sample weight due to furnace heating gives a mechanical elongation or contraction of the springs. The LVDT core, or armature, also moves to give a proportional electrical signal. The signal is electro-mechanically demodulated by a conventional chopper network and fed in dc form into the recorder Y^1 axis (Hewlett/Packard Model 136A). During calibration, the core must be closely positioned at the null point, or electrical center. This is the purpose of the micro adjuster shown on the functional diagram (Figure 3).

A Pyrex ball joint serves as a porthole to the calibration pan that is attached to the main support rod. The pan has a twofold purpose. Tare weights give the correct spring stretch when different sample or crucible weights are used for a run. The pan also catches the weights dropped in the calibration pan. This small ring is submerged in the oil pool. Random spring oscillations from normal vibrations or sudden weight change are thus kept to a minimum.

A thermocouple sheath is fastened by a metal pin to the hooked support rod at a point just below the oil pool. The three 0.010 inch diameter thermocouple wires are each connected by a small piece of stainless steel tubing to one end of a tungsten filament. The other end of the filament is similarly crimped with the crimped tube resting in a mercury pool. Electrical continuity through the Pyrex glassware is provided by tungsten rod feedthroughs. Outside the glassware, one electrode is grounded. Paired compensated leadwire runs directly to an ice bath with copper wire then feeding into the recorder X axis

through an input filter (Hewlett/Packard Model 17106A). The filter is carefully tuned with a sinusoidal generator and oscilloscope for maximum rejection of 60 cps noise.

The sample crucible is radially connected by a pin to the thermocouple sheath at the bottom of the sheath. The crucible, pin and sheath are all made from recrystallized alumina (McDanel Refractory Porcelain Company, Body AP35).

Operational reproducibility has been good. The phenolic resin of Figure 25 is run each time the thermocouple is replaced with maximum weight deviations not exceeding ±1% at any temperature*. Further, the actual and plotted weights from weighing the sample at the end of the run usually agree within ±0.5%. Handling and room humidity are factors in this error. The possible magnitude of weight change of polyaromatic resins and refractory reinforcements to high temperatures is revealed upon comparison of a thermogram for a carbon cloth reinforced polybenzimidazole (PBI) with those for a conventional phenolic resin and carbon cloth reinforced phenolic resin (Figures 25, 26, 27). These materials are further described by Table III.

There are three additional primary thermobalance systems: furnace, furnace tube, controller and recorder; the vacuum/purge system; and the time base generator and interruptive marker. Each of these and the suspension system will now be reviewed in detail with emphasis on modifications made to supplement the original Thermo-Grav. Commercial apparatus other than Aminco is generally identified by trade name and model number.

*Table 1 permits a more precise conversion of thermogram X axis deflection to temperature in ^{o}C or ^{o}K.

III. TWO PEN RECORDER

A. GENERAL

The Hewlett/Packard, Moseley Division, Model 136AR-02 recorder shown in Figure 4 is designed to plot two coordinate curves from dc signals representing a single independent variable (in this case - temperature) and two dependent variables (in this case - time and weight). The two pens move vertically on a single carriage which travels horizontally. Each pen has a full scale plotting range in both vertical and horizontal directions. The recorder platen accepts 8-1/2 x 11 inch graph paper. Each axis is controlled by an independent servo mechanism with one megaohm input resistance at null on eleven variable ranges.

B. X AXIS - TEMPERATURE

The recorder carriage or X axis is driven by the emf output of the thermocouple imbedded in the sample. Because the readout is linear volts, it is necessary to calibrate in terms of volts and convert the volts-readout to degrees by use of a standard conversion table. In order to facilitate temperature determination, the graph range was "stretched" to twenty inches by repeating the sweep when the carriage reached the ten inch mark on the graph. This is easily accomplished by quickly turning the X zero control to reposition the carriage to the X axis zero when the thermocouple emf has driven the carriage to the ten inch position. With the platinum, platinum+ 13% rhodium thermocouple, the conversion table emf at 1400°C is 15.969 mv. Using 16.000 mv as the basis for full scale (twenty inches), then 8.000 mv is equal to ten inches, or about 806°C. There is, therefore, one sweep from room temperature to 806°C and a second sweep from 806°C to 1400°C. For most of the testing done so far, 1400°C has been the high point, but this recording technique is easily adaptable to many ranges with this recorder. Starting at room tempe_ature is accomplished by shorting the X axis input at the ice bath and adjusting the carriage position with the X zero to indicate zero inches. When the short is removed, the pen indicates the millivolts equivalent (by con ersion) to room temperature.

Calibration of the X axis is accomplished by converting the high point temperature to millivolts and rounding off to some easy-to-use figure (1400°C = 15.969 mv, round off to 16.000 mv). This figure is then divided by two giving the ten inch calibration voltage

required. The X axis input is then shorted upstream of the input filter. The filter will affect the calibration span and must be used when calibrating. The carriage is then positioned by the X zero control to indicate zero inches. The short is removed and a dc voltage (the required calibration voltage) is applied. The variable range control is then adjusted to position the carriage to indicate ten inches. With the calibration voltage applied, the X zero control is used to reposition the carriage to again indicate zero inches. The final voltage (in the case of 1400°; 16 mv) is applied as a check for proper indication of ten inches again (twenty inches total carriage travel).

C. Y^1 AXIS - WEIGHT

The Y^1 axis of the recorder is used to indicate sample weight change. The proportional ac signal from the linear variable differential transformer is fed to a demodulator circuit where it is mechanically converted to dc and filtered. From the demodulator a shielded cable carries the signal to the recorder Y^1 axis. The Y^1 pen, carried on the X carriage, is then positioned to indicate a relative weight at a given temperature.

Weight calibration is accomplished by adjusting pen travel when accurate calibration weights are added to the suspension system. It should first be determined approximately what percentage of its weight the sample will gain or lose. By calibrating for this percentage, a broader, easier to decipher graph results. For instance, if a material contained 60% free carbon, it could probably be safely assumed that the material would not lose more than 50% of its weight at temperatures below 1400°C. Therefore a 40 mg sample could allow a 4 mg/inch calibration (20 mg/5 inch). To accomplish this, the sample is weighed into the crucible and the crucible is attached to the suspension system. The system envelope is closed and sealed with the exception of the calibration access port. Only the port remains open to minimize the effects of air currents on the suspension system. With the LVDT disconnected, the Y^1 pen is positioned at the center of the graph Y axis by the Y^1 zero control. The LVDT is reconnected and a weight, one half of the desired calibration span, is placed on the calibration pan. By means of the LVDT micro adjuster, the LVDT is moved to position the Y^1 pen at the center of the graph Y axis. The LVDT null now equals the recorder null and the LVDT should not again be moved. Another weight is then placed on the calibration pan, so that the total weight equals the desired span. The Y^1 pen is now positioned by the Y^1 variable range control to the point on the Y axis

that will indicate starting weight if a weight loss is expected or allow for maximum weight gained if a gain is expected. Removal of both weights should cause the pen to travel the length of the desired Y axis span. When sample weight loss is expected, the weights should be left on the calibration pan after completion of the calibration. This allows the Y^1 pen to travel down the length of the Y axis as the sample loses weight.

D. Y^2 AXIS - TIME

The Y^2 pen of the recorder is used to indicate elapsed time during a run. This indicates run reliability and allows accurate replotting of heating rates. The output of the time base generator is fed directly to the Y^2 input. During a run, the Y^2 pen, forced to move vertically by the time base generator and horizontally by the sample temperature, plots a graph of time versus temperature.

When calibrating the Y^2 axis for time, zero minutes is manually set on the digital readout and the Y^2 pen is positioned on the Y axis zero inches line by the Y^2 zero control. The nearest maximum time, above desired time, is set on time base voltage divider selector switch. The desired length of time is then set on the digital readout ("minutes", recorder X axis section) on the front panel. Owing to each recorder range being variably controlled, there is an infinite number of available time bases that can be used. The figures in Table 2 show most of the available ranges in the "fixed" mode. The fixed 50 mv/inch range satisfies most requirements. Setting the Thermo-Grav panel digital readout moves the Y^2 pen according to time base setting. Precise pen positioning for a given "final" time can be made by the time base "fine adjust" control. After adjusting for proper pen travel for a given time, the digital readout is reset to zero. At the start of the run, the "seconds-minutes" switch is moved to the proper mode, starting the time base unit.

IV. SAMPLE TEMPERATURE MONITORING

A. THERMOCOUPLE/CRUCIBLE ASSEMBLY

Sample temperature is monitored by a 3-wire platinum, platinum+13% rhodium, platinum (ground) thermocouple (Figure 5). A third leg was added to the conventional two wire thermocouple when it was found that a great deal of random noise was coupling to the thermocouple signal at temperatures above about 1000°C. This unwanted noise was adding to the true thermocouple emf resulting in an erroneous sample temperature indication. By actually grounding the thermocouple bead with a separate platinum lead, most of the unwanted signal is diverted to ground leaving the true signal relatively "clean". Any 60 cycle noise remaining is rejected by a filter just before entering the recorder circuitry. The two major leads are shielded by an alumina sheath with the third lead running up the outside (an identical sheath but containing the necessary three holes has been ordered). The sheath also serves as the support for the crucible (Figure 6). A 0.039 inch hole, 1/4 inch from each end of the 13-1/2 inch long sheath serves as a means of attaching both the crucible to the sheath and the sheath to the Pyrex main support rod. The crucible walls also have two opposite 0.039 inch holes located near the upper edges. A 0.035 inch alumina pin passes through the crucible walls and through the hole in the bead end of the sheath. Approximately 1/4 inch of the thermocouple bead is exposed, allowing it to be placed directly into the sample contained in the crucible.

The sheath is connected to the Pyrex main support rod by a wire pin. The lower Pyrex support end is formed in the shape of two "stirrups" to accept the pin (Figure 7). The three thermocouple leads extend 5/8 inch beyond the end of the sheath. The leads are bent over and away from the sheath in a manner similar to umbrella stays. Each lead is connected to a 1 inch long, 0.0005 inch tungsten filament by means of a short crimped piece of stainless steel capillary tubing. The other end of the filament is similarly crimped to add weight to the filament for firm contact on the surface of a pool of mercury.

B. MERCURY POOLS

There are two major mercury pools. They are electrically isolated from each other by a glass partition to accept the negative and positive thermocouple leads (Figures 7 and 8). A tungsten rod electrode extends through the Pyrex glassware wall and into each

pool. Attached to each electrode is the proper platinum or platinum-rhodium compensated lead wire which terminates in a glass tube of mercury in an ice bath. Copper wires complete the last leg of the circuit between the mercury filled glass tubes and the Hewlett/Packard Model 17106A filter which is plugged directly into the recorder X axis input.

A third, much smaller mercury pool is contained in the formed open end of a glass tube that rests inside the vacuum adapter (Figure 8). A tungsten electrode extends from this pool through a glass partition out the end of the tube. This electrode is grounded to the metal support column by a short piece of copper wire.

To minimize the mercury evaporation, the glassware surrounding the mercury pools is kept below room temperature by water cooling. Soft copper tubing surrounds the glassware and is held tight against it with a large hose clamp (Figure 7). Cold water flows through this tubing at all times.

V. FURNACE SYSTEM

A. FURNACE

The furnace is manufactured by Tem-Pres Research, Inc. (Model SQ-IC-4). Being of the dual construction type, it permits access to the center chamber for forced air blower cooling after use (Figure 9). The spherical center chamber is 4 inches in diameter. There is a uniform hot zone of some 2 inches diameter at geometric center, excluding furnace tube end losses. A slightly larger than one inch OD access port extends from the upper half of the furnace into the chamber. This port accepts the one inch OD alumina furnace tube. A platinum+40% rhodium, 0.030 inch diameter internally wound wire element and alumino-silicate fiber thermal insulation serve to give a practical furnace working temperature of 1600°C with limited service to 1800°C. Two parallel furnace pedestals with knurled set screw holders permit movement of either half or the complete furnace along a vertical length of approximately 10 inches. This allows precise placement of the furnace hot zone around the crucible. The outer shell of the furnace is partially water cooled to minimize temperature increases inside the cabinet during operation.

B. CONTROLLER AND RECORDER

Both isothermal and programmed heating rates are controlled by a modified F&M Model 240M Power Proportioning Temperature Programmer (Figure 10). Modifications made by the manufacturer to specifications include (a) a line input of 208 vac at 25 amps, (b) power output of 0-5 KVA, (c) addition of a 4 rpm synchronous timing motor to supply eleven more heating rates to as high as 600°C/min, (d) several minor internal modifications.

Although the unit was designed to operate from a Chromel/alumel thermocouple, it is forced to use the signal from the platinum, platinum+10% rhodium thermocouple of the Tem-Pres furnace. This has affected the linearity of the programmed heating rates only slightly. For a nominal heating rate of 8.5°C/min, the averaging tangents drawn along the limits of the heating rate curves of 2 runs bracketing a minimum and maximum heating rate reproducibility of 9 runs show a deviation of less than ±1°C/min (Figure 11).

The heating rate of the furnace is monitored by a Leeds and Northrup potentiometric strip chart recorder connected directly to the same thermocouple leads as the controller . The recorder has a range of 0-1600°C with a chart travel of 20 min/inch.

C. FURNACE TUBE

The one inch OD, 9-1/2 inch long furnace tube is fabricated of McDanel Body AP35 recrystallized alumina so that it can withstand furnace temperatures up to 1800°C. The tube is closed at one end and fitted with a flat-surface aluminum flange at the other (Figure 12). The tube is bonded to the flange with Hysol Epoxi-Patch. The flange is fitted with a brass hose nipple that also holds the high vacuum rubber tubing for the vacuum/purge system.

To ensure a vacuum tight seal between the furnace tube flange and lower glassware flange (just below the mercury pools), an "O" ring is used. The Buna-N rubber "O" ring, slightly smaller in outside diameter than the flange, is held by a stainless steel reflector plate. The center plate hole is just large enough to allow the crucible to pass through when lowering the furnace tube. This plate is thinner than the "O" ring to permit the ring to collapse slightly and form a seal between the two flanges. Apiezon vacuum grease Type T assures a good seal. The plate also acts to reflect much of the heat back inside the furnace tube. No. 1, 4 inch spring clamps are used to hold the two flanges together with the "O" ring sandwiched between. Four of these clamps, equally spaced around the flange, also act as heat radiators and keep the temperature of the aluminum flange below 50°C.

VI. TIME BASE GENERATOR

A. GENERAL

The time base generator consists of (a) a regulated 12 vdc power supply, (b) a Standard Type 152 Dual Motor system, (c) the original Aminco Time Drive assembly with drive motor removed, and (d) an adjustable, 5-step voltage divider.

Briefly, the regulated 12 vdc is applied across a voltage divider containing a precision 10 turn potentiometer. A linear voltage increase is picked off the potentiometer and applied to the recorder. The potentiometer is automatically rotated at a preselected rate of either one turn every 40 min (1/40 rpm) or one turn every 40 sec (1-1/2 rpm) to a maximum of 10 turns (400 min or sec).

B. POWER SUPPLY

The power supply is constructed from Figure 10.9 in the Seventh Edition of the General Electric Transistor Manual. This supply is adequate and neither difficult nor expensive to assemble.

C. TIME DRIVE ASSEMBLY

The Standard Type 152 Dual Motor is available from Giannini Controls Corporation. The unit used has a 1/4 inch diameter, one inch long output shaft. Shaft speeds are 1/40 rpm and 1-1/2 rpm (counter-clockwise). This unique assembly bolts quite nicely to the back of the existing Aminco Time Drive assembly using one inch spacers (Figure 13). The motor in the Aminco unit is removed. The slip-clutch and gear on the motor shaft are now on the Dual Motor output shaft. They are aligned to mesh with the drive gear for an existing 5 K ohm precision potentiometer.

The leads for each motor are run to a center-off, double-throw switch. This allows immediate selection of either minutes or seconds and a start from zero at the beginning of the run.

D. VOLTAGE DIVIDER

The simple but effective voltage divider (Figure 14) consists of a 6-position rotary switch, 5 precision fixed resistors, a 1-turn

potentiometer and a precision 10-turn potentiometer. The latter is a part of the original Aminco Time Drive assembly. When the time base selector switch is moved to a position other than "Off", current flows through the precision potentiometer. The amount of current that flows depends on the total resistance of the circuit as dictated by the selector switch position. This current can be finely adjusted by the 1 K ohm potentiometer.

For example, with the selector switch at the 5 position, there is a total resistance of approximately 15 K ohms (fine adjust potentiometer at 1/2 turn). This gives a voltage drop of about 4 volts across the 5 K ohm precision pot. With the time drive assembly rotating this pot at a rate of 1/40 rpm, the pot slidewire will go positive (in reference to the indicated negative output) at a rate of 10 mv/min. With the recorder at a range of 50 mv/inch, the pen will travel at a rate of 5 min/inch.

VII. INTERRUPTIVE MARKER

The circuit is basically the same as the original Aminco unit with the exception of the two diodes and the addition of a "preset" switch (Figure 15). The switch allows pre-setting of marker height. This circuit "steals" a 6 vdc operating power from a bridge across the regulated 12 vdc power supply used in the time base generator. The original Aminco cam micro switch assembly and minutes-per-mark switch are retained. The capacitor is charged thru D_2. When S_1 closes, the capacitor discharges, passing a positive spike through D_1. The original type IN35 diode allowed some signal to pass D_1 when the capacitor was charging. This negative signal caused an unwanted "blip" on the chart. The new type IN3595 diodes have a much higher back resistance, eliminating this negative spike completely.

VIII. SUSPENSION SYSTEM

A. VERTICAL MICRO ADJUSTER

It is often necessary to move the entire suspension system up or down using the vertical micro adjuster (Figures 16 and 17). The knurled knob of the micro adjuster is internally threaded (40 threads/inch) and held in place by a right-angle support. A slotted, threaded shaft passes through the knob and supports the springs. The shaft is keyed to prevent secondary rotation. Any knob movement moves the shaft and entire suspension system vertically at the rate of 0.025 inch/revolution. A modified valve collar is threaded into the base of the ball joint-shaped body. An "O" ring provides a vacuum tight seal. Another "O" ring held in place by a packing cap seals the shaft like a valve stem. The body also holds a 1/8 inch Swagelok fitting which in turn provides support and a vacuum tight seal for the spring housing temperature thermocouple.

B. LVDT MICRO ADJUSTER

The LVDT coil of the original Thermo-Grav was manually positioned for an electrical null point during calibration. This gave highly erratic pen movement during calibration of the sensitive Y^1 axis of the new recorder. Further it was suspected that the old coil holder may have slipped on several occasions. Therefore, an LVDT micro adjuster was made to aid in calibration and provide firm support of the coil (Figures 18 and 19). Clamp A of this device is firmly bolted around the coil. Two threaded shafts (40 threads/inch) occupy each side. The shafts mate with the threaded legs of Fork B. Springs C exert sufficient pressure to give the desirable smooth movement of Clamp A when the two knurled knobs are rotated together. The rate is 0.025 inch/revolution. Fork B is rigidly bolted to Mount D which in turn bolts to a heavy metal column in the Thermo-Grav cabinet. Elongated screw holes allow correction for LVDT coil and glassware, suspension system, or other minor misalignment. Mount D normally remains in place and the rest of the assembly is taken down for glassware removal. The entire adjuster is made from aluminum to reduce possible interference with the LVDT inductive field.

C. CONSTANT TEMPERATURE WATER BATH

The precision springs are sensitive to ambient room temperature changes. The use of a controlled temperature spring housing and

water bath adopted to reduce this effect can be illustrated by the procedure for finding the best bath operating temperature. The spring housing temperature was controlled by a constant temperature water circulator (Precision Scientific Company Catalog No. 66600). The Y^1 recorder axis was 4 mg/inch; any pen change came from ambient temperature change. The spring chamber thermocouple was fed into the existing Thermo-Grav sample temperature panel readout counter. The Y^1 pen moved across the chart via a time base signal applied to the X carriage axis. Figure 20 shows results for water baths from room temperature to 65°C. The best bath temperature was then set (54°C). It is visually monitored during runs using the sample temperature readout. A strip chart has also been used for a permanent record. Room temperature changes of 5°C have little effect on either chamber temperature or sample weight with a controlled temperature spring housing. In contrast, a 5°C ambient change has given a weight error of as much as 4 mgm without the water bath. Other unknown parts of the thermobalance are also ambient temperature sensitive. Therefore, every effort is made during a run to maintain constancy. This includes the use of two cooling blowers to dissipate excessive furnace and spring housing heat that builds up in the partially enclosed Thermo-Grav cabinet.

IX. VACUUM/PURGE SYSTEM

It is often desired to observe the effects of different atmospheres on a sample under test. This is accomplished by the vacuum/purge system (Figures 21, 22 and 23). When a vacuum atmosphere is desired, the entire system envelope can be brought down to internal pressures as low as 10 microns of mercury by a Cenco Model Megavac mechanical vacuum pump. The pressure is constantly monitored by a thermocouple tube vacuum gauge with the tube mounted by a short piece of rubber tubing on the vacuum adapter. Another thermocouple tube is mounted on the system manifold. A switch selects the readout of either of these two tubes to simplify the search for leaks by isolation.

When it is desired to fill the envelope with a gas such as helium, the envelope is first evacuated by closing Valves A and B and opening valve C (Figure 21). Valve A is then opened, with the vacuum pump continuing to run, pulling the gas through the envelope for several seconds. Valve A is then closed and the envelope is again evacuated. This procedure is repeated three times. The third time, instead of closing valve A, Valve C is closed stopping the flow of gas out of the envelope. The vacuum pump is turned off and valve B is opened to allow that part of the manifold to return to normal atmospheric pressure. The envelope is allowed to fill with the gas to just slightly above atmospheric pressure as read on the Bourdon gauge. At this point, valve C is reopened allowing a path of flow for the gas out of the system.

Gas pressure is controlled at the gas bottle manifold (Figure 23) by a pressure regulator. Line pressure is kept at 20 psi. Flow is monitored by a flowmeter with tubes interchangeable with different gases. Any surges in flow are damped out by a 36 inch length of capillary tubing upstream of the envelope. The gas bottle manifold allows any one of three different types of gas to be used without manually switching bottles.

Helium is most often used as an atmosphere inside the envelope because it has the lowest density of the inert gases. Lower density gases considerably decrease convection currents inside the envelope, thereby decreasing suspension system "bounce" and producing an easier to decipher sample weight change thermogram.

X. SAMPLE PREPARATION

A. MACHINE LATHE

A unique sample preparation technique yields a fine powder from bulk material through use of a machine lathe. The method permits uniform particle distribution for samples of cloth reinforced plastics that have poor grinding qualities by any other technique. The bulk sample is held in a three or four jaw chuck depending upon size and configuration. The chuck and sample are rotated at a slow rate of 52 rpm. A highly sharpened, carbide cutting tool is drawn across the face of the sample at a rate of 0.0027 inch/revolution and a depth-of-cut of 0.001 inch. Due to the various resin to reinforcement relationships, all materials do not yield the same powder configuration. Each batch is inspected through a microscope and grain size is measured with a calibrated ocular to aid in maintaining a relatively constant yield shape and size. When a batch shows poor ratios of resin/reinforcement or exceptionally large or small grains, the sample position in the chuck is changed. At times the yield is better cutting across the opposite plane of the sample cloth. Occasionally a sample must be glued to a disc and the disc clamped in the chuck (Figure 24). This permits a more perpendicular cut and usually changes the yield configuration.

B. SIEVING AND DRYING

After a sufficient quantity of material is cut, it is sieved to -325 sieve (0.0017 inch diameter grain size) and reinspected. If the quantity of waste material that did not pass through the sieve is not great and the yield seems to show a good resin/reinforcement ratio it is then weighed. After weighing the yield is dried for 50 minutes at 125°C at a reduced pressure of approximately 18 inches of Hg and then again weighed. Comparison between the two weights is an indication of free moisture content.

XI. CLEANING OF ALUMINA COMPONENTS

A. AIRBRASIVE UNIT

Each time the thermocouple is replaced, the sheath is thoroughly cleaned. Minute quantities of hard deposit sometimes form on the sheath. Because these deposits are in very small percentage for each run, they do not introduce an appreciable weight error. The deposit is difficult to remove by normal cleaning procedures. When this happens, the sheath is cleaned by blasting it with aluminum oxide powder at 80 psi in an S. S. White Company Airbrasive unit. This unit has served well for cleaning the sheath, crucible, and furnace tube. There is roughly 0.1% of alumina removed each time a part is cleaned in the Airbrasive unit but this does not seem to affect the usefulness of the part. After blasting, the part is cleaned with Lava soap and water to remove all traces of the oxide.

B. SOAP AND WATER

The same crucible is used for every run necessitating a thorough cleaning each time. In most cases, a good scrubbing with Lava handsoap is sufficient. The pumice in this soap works nicely to remove the residue without affecting the alumina. Rinsing in wood alcohol then distilled water removes all traces of soap. The crucible is then dried by holding in a gas flame several seconds. The weight of the crucible in monitored to the nearest 0.1 mg to indicate if any residue remains. At times it is necessary to clean the crucible in the Airbrasive unit due to a heavy or exceptionally hard residue.

XII. FURTHER WORK

A. SAMPLE TEMPERATURE MONITORING

The recording thermobalance has a major problem of a short, usable sample thermocouple life. Depending on the sample material, the thermocouple can only be relied on for a maximum of 8 or 9 runs to 1400°C. Some materials apparently react with the exposed thermocouple bead with serious deterioration. Runs have been made to 1600°C but the random noise level above 1500°C makes it difficult to determine the true temperature. The platinum, platinum+13% rhodium thermocouple is only good for one run to 1600°C before it becomes inoperative. For these reasons, other types of bare wire or sheathed thermocouples are being considered to increase the usable time between sample thermocouple replacement.

In order to protect the third or ground sample thermocouple lead, a special 3-hole sheath is being fabricated by the McDanel Refractory Company. According to the company, this new sheath will be the same length and diameter of the old part but will be slightly heavier.

B. RADIANT FURNACE SYSTEM

Future plans call for the installation of a Research, Inc., Dual Elliptical Reflector furnace using two tungsten filament, quartz tube radiant heating lamps. This furnace, together with a Vycor furnace tube and the F&M programmer, is expected to give unique constant heating rate and isothermal capabilities to about 1000°C.

C. VACUUM/PURGE SYSTEM

Although the present system conveniently gives a high purity envelope atmosphere, many parts incur leaks and other failures frequently. The system is due for a revamping using more functional and modern components although the basic flow patterns will remain the same.

D. SPECIAL MATERIALS STUDIES

Several topics are of interest that may require minor changes to the recording thermobalance. These relate to a comparison of results for the slow constant heating rate case with those for a better

approximation of aerospace environments. Possible examples include constant heating rates to high values, isothermal thermogravimetry with a minimum time to reach the set point temperature, sample size effects, and similar work.

TABLE I

THERMOGRAM X-AXIS DEFLECTION/TEMPERATURE CONVERSION

Deflection, inch	Temperature, °C	Temperature, °K	Deflection, inch	Temperature, °C	Temperature, °K
0.0	0.0	273.16	4.0	380.6	653.76
0.1	14.4	287.56	4.1	388.4	661.56
0.2	28.0	301.16	4.2	396.2	669.36
0.3	41.0	314.16	4.3	404.0	677.16
0.4	53.5	326.66	4.4	411.7	684.86
0.5	65.5	338.66	4.5	419.4	692.56
0.6	77.1	350.26	4.6	427.0	700.16
0.7	88.4	361.56	4.7	434.6	707.76
0.8	99.3	372.46	4.8	442.2	715.36
0.9	109.9	383.06	4.9	449.8	722.96
1.0	120.3	393.46	5.0	457.3	730.46
1.1	130.4	403.56	5.1	464.9	738.06
1.2	140.3	413.46	5.2	472.4	745.56
1.3	150.1	423.26	5.3	479.9	753.06
1.4	159.9	433.06	5.4	487.4	760.56
1.5	169.5	442.66	5.5	494.9	768.06
1.6	178.8	451.96	5.6	502.3	775.46
1.7	188.1	461.26	5.7	509.7	782.86
1.8	197.2	470.36	5.8	517.0	790.16
1.9	206.3	479.46	5.9	524.3	797.46
2.0	215.2	488.36	6.0	531.6	804.76
2.1	224.1	497.26	6.1	538.9	812.06
2.2	232.9	506.06	6.2	546.1	819.26
2.3	241.6	514.76	6.3	553.3	826.46
2.4	250.2	523.36	6.4	560.5	833.66
2.5	258.7	531.86	6.5	567.6	840.76
2.6	267.2	540.36	6.6	574.8	847.96
2.7	275.6	548.76	6.7	582.0	855.16
2.8	284.0	557.16	6.8	589.1	862.26
2.9	292.3	565.46	6.9	596.2	869.36
3.0	300.5	573.66	7.0	603.2	876.36
3.1	308.7	581.86	7.1	610.3	883.46
3.2	316.8	589.96	7.2	617.2	890.36
3.3	324.9	598.06	7.3	624.2	897.36
3.4	333.0	606.16	7.4	631.2	904.36
3.5	341.0	614.16	7.5	638.1	911.26
3.6	349.0	622.16	7.6	645.1	918.26
3.7	356.9	630.06	7.7	652.0	925.16
3.8	364.9	638.06	7.8	659.0	932.16
3.9	372.8	645.96	7.9	665.9	939.06
4.0	380.6	653.76	8.0	672.7	945.86

TABLE I (Cont.)

Deflection, inch	Temperature, °C	Temperature, °K	Deflection, inch	Temperature, °C	Temperature, °K
8.0	672.7	945.86	12.0	933.2	1206.36
8.1	679.6	952.76	12.1	939.4	1212.56
8.2	686.4	959.56	12.2	945.6	1218.76
8.3	693.2	966.36	12.3	951.9	1225.06
8.4	700.0	973.16	12.4	958.0	1231.16
8.5	706.8	979.96	12.5	964.1	1237.26
8.6	713.4	986.66	12.6	970.2	1243.36
8.7	720.2	993.36	12.7	976.3	1249.46
8.8	727.0	1000.16	12.8	982.4	1255.56
8.9	733.7	1006.86	12.9	988.5	1261.66
9.0	740.4	1013.56	13.0	994.6	1267.76
9.1	747.1	1020.26	13.1	1000.7	1273.86
9.2	753.7	1026.86	13.2	1006.8	1279.96
9.3	760.3	1033.46	13.3	1012.8	1285.96
9.4	766.9	1040.06	13.4	1018.9	1292.06
9.5	773.5	1046.60	13.5	1024.9	1298.06
9.6	780.1	1053.26	13.6	1030.8	1303.96
9.7	786.6	1059.76	13.7	1036.8	1309.96
9.8	793.2	1066.36	13.8	1042.8	1315.96
9.9	799.7	1072.86	13.9	1048.7	1321.86
10.0	806.2	1079.36	14.0	1054.6	1327.76
10.1	812.7	1085.86	14.1	1060.5	1333.66
10.2	819.2	1092.36	14.2	1066.4	1339.56
10.3	825.6	1098.76	14.3	1072.3	1345.46
10.4	832.1	1105.26	14.4	1078.2	1351.36
10.5	838.5	1111.66	14.5	1084.1	1357.26
10.6	844.9	1118.06	14.6	1089.9	1363.06
10.7	851.3	1124.46	14.7	1095.8	1368.96
10.8	857.7	1130.86	14.8	1101.7	1374.86
10.9	864.1	1137.26	14.9	1107.6	1380.76
11.0	870.5	1143.66	15.0	1113.4	1386.56
11.1	876.8	1149.96	15.1	1119.2	1392.36
11.2	883.1	1156.26	15.2	1125.1	1398.26
11.3	889.4	1162.56	15.3	1130.9	1404.06
11.4	895.7	1175.16	15.4	1136.8	1409.96
11.5	902.0	1181.36	15.5	1142.6	1415.76
11.6	908.2	1187.66	15.6	1148.4	1421.56
11.7	914.5	1187.66	15.7	1154.2	1427.36
11.8	920.7	1193.86	15.8	1160.0	1433.16
11.9	927.0	1200.16	15.9	1165.8	1438.96
12.0	933.2	1206.36	16.0	1171.5	1444.66

TABLE I (Cont.)

Deflection, inch	Temperature, °C	Temperature, °K
16.0	1171.5	1444.66
16.1	1177.3	1450.46
16.2	1183.1	1456.26
16.3	1188.9	1462.06
16.4	1194.7	1467.86
16.5	1200.5	1473.66
16.6	1206.2	1479.36
16.7	1212.0	1485.16
16.8	1217.8	1490.96
16.9	1223.5	1496.66
17.0	1229.3	1502.46
17.1	1235.1	1508.46
17.2	1240.8	1513.96
17.3	1246.6	1519.76
17.4	1252.3	1525.46
17.5	1258.1	1531.26
17.6	1263.8	1536.96
17.7	1269.6	1542.76
17.8	1275.4	1548.56
17.9	1281.1	1554.26
18.0	1286.9	1560.06
18.1	1292.6	1565.76
18.2	1298.4	1571.56
18.3	1304.1	1577.26
18.4	1309.9	1583.06
18.5	1315.7	1588.86
18.6	1321.4	1594.56
18.7	1327.2	1600.36
18.8	1333.0	1606.16
18.9	1338.7	1611.86
19.0	1344.5	1617.66
19.1	1350.3	1623.46
19.2	1356.0	1629.16
19.3	1361.8	1634.96
19.4	1367.6	1640.76
19.5	1373.4	1646.56
19.6	1379.1	1652.26
19.7	1384.9	1658.06
19.8	1390.7	1663.86
19.9	1396.4	1669.56
20.0	1402.2	1675.36

Thermogram X-Axis calibration of 0.8 millivolt/inch deflection and a Platinum, Platinum 13% Rhodium thermocouple with reference junctions at 0.0°C. Shenker, H., et al, "Reference Tables For Thermocouples," NBS Circular 561, 27 April 1955, Table 3 page

TABLE II
AVAILABLE TIME BASES

Recorder Range	Time Base Range - Minutes				
	1.25 mv/min	2.5 mv/min	5 mv/min	10 mv/min	25 mv/min
.5 mv/in	.4 min/in	.2 min/in	.1 min/in	.05 min/in	.02 min/in
1 mv/in	.8 min/in	.4 min/in	.2 min/in	.1 min/in	.04 min/in
5 mv/in	.4 min/in	2 min/in	1 min/in	1 min/in	.2 min/in
10 mv/in	8 min/in	4 min/in	2 min/in	5 min/in	.4 min/in
50 mv/in	40 min/in	20 min/in	10 min/in	10 min/in	2 min/in
.1 v/in	80 min/in	40 min/in	20 min/in	50 min/in	4 min/in
.5 v/in	400 min/in	200 min/in	100 min/in	100 min/in	20 min/in
1 v/in	800 min/in	400 min/in	200 min/in		40 min/in
	Time Base Range - Seconds				
	1.25 mv/sec	2.5 mv/sec	5 mv/sec	10 mv/sec	25 mv/sec
.5 mv/in	.4 sec/in	.2 sec/in	.1 sec/in	.05 sec/in	.02 sec/in
1 mv/in	.8 sec/in	.4 sec/in	.2 sec/in	.1 sec/in	.04 sec/in
5 mv/in	4 sec/in	2 sec/in	1 sec/in	.5 sec/in	.2 sec/in
10 mv/in	8 sec/in	4 sec/in	2 sec/in	1 sec/in	.4 sec/in
50 mv/in	40 sec/in	20 sec/in	10 sec/in	5 sec/in	2 sec/in
.1 v/in	80 sec/in	40 sec/in	20 sec/in	10 sec/in	4 sec/in
.5 v/in	400 sec/in	200 sec/in	100 sec/in	50 sec/in	
1 v/in	800 sec/in	400 sec/in	200 sec/in	100 sec/in	

TABLE III
EXPERIMENTAL MATERIALS

Code:	RI 4009	9-35-C	N151-35-C
Description:	phenolic resin	phenolic/carbon cloth	polybenzimidazole/ carbon cloth
Cloth Reinforcement:			
Number of plies	none	52	60
Style		square weave	square weave
Cure Cycle:			
Pressure, psi	320	500	500
Time, hr at each Temperature, °C	0.5-92; 0.5-160	0.08-149; 3-149	0.33-371; 3-371
Postcure Cycle:			
Time, hr at each Temperature, °C	24-149; 24-177	----[d]	----[e]
Test Laminates:			
Barcol Hardness	0	70	70
Dimensions, inch	1 x 4 x 0.125	0.75 diameter x 0.50	3.35 x 2 x 0.5
Resin Content, %[a]	100	37.9	37.4
Specific Gravity	1.18	1.38	1.34
Void Content, %[b]	nil	22.3	29.7
Trade Names[c]:			
Reinforcement		CCA-1	CCA-1
Resin	RI 4009	CTL-91LD	Imidite 2803

[a] By weight percent of resin

[b] By volume percent of voids in resin

[c] CCA-1 (H. I. Thompson Fiberglas Company)
CTL-91LD (CTL, Incorporated)
Imidite 2803 (Narmco Materials Division
RI 4009 (Monsanto Chemical Company)

[d] 18 hr at 135°, 72 hr from 135° to 204°, 4 hr at 204°, 7 hr cooling to below 93°.

[e] 26 hr from room temperature to 316°, 24 hr at 316°, 10 hr from 316° to 343°, 24 hr at 343°, 10 hr from 343° to 371°, 24 hr at 371°, 10 hr from 371° to 399°, 6 hr at 399°, 10 hr from 399° to 427°, 6 hr at 427°, 10 hr from 427° to 454°, 6 hr at 454°, 10 hr cooling to below 93°. Postcured in an argon atmosphere.

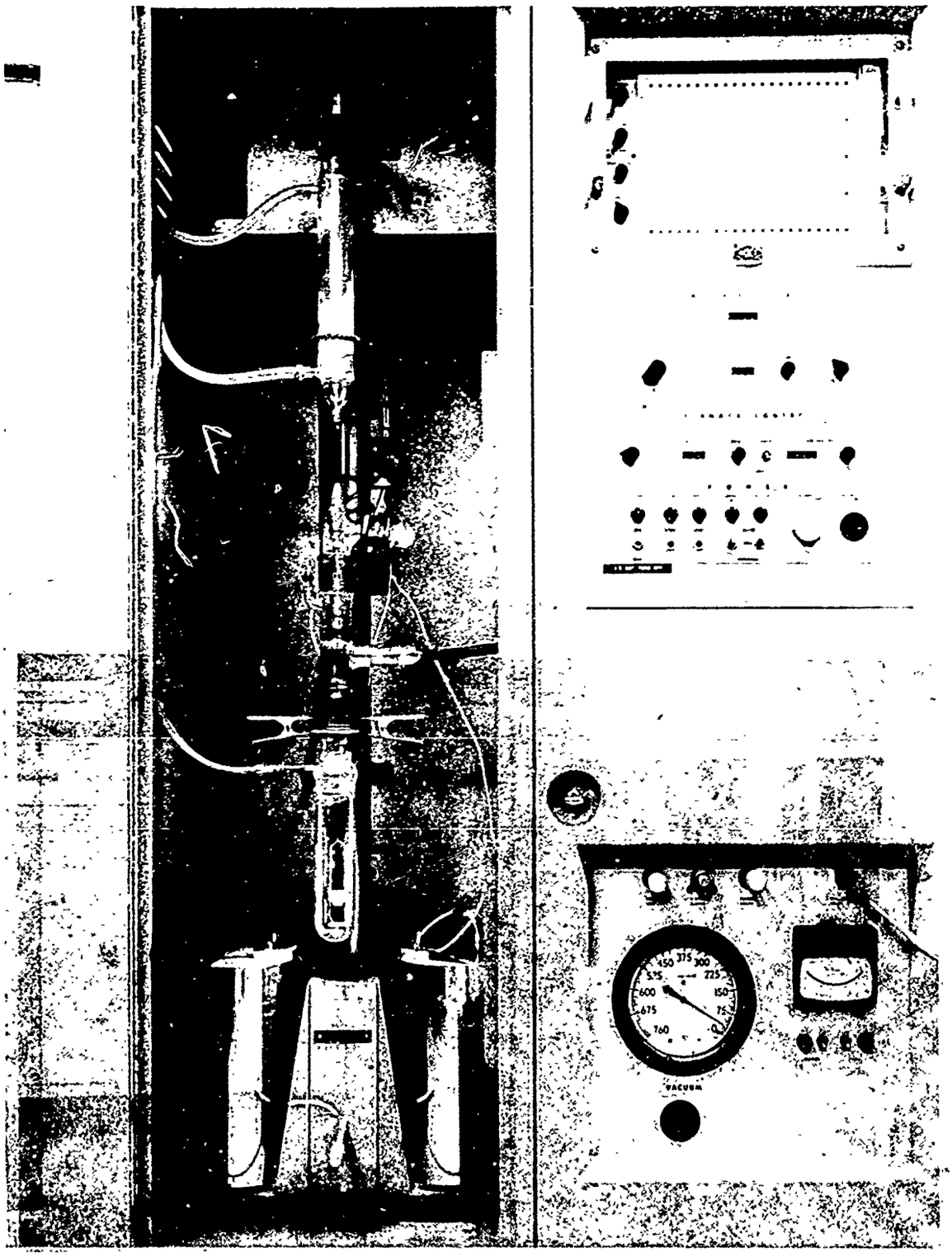

Figure 1. General View of the Recording Thermobalance Before Modification

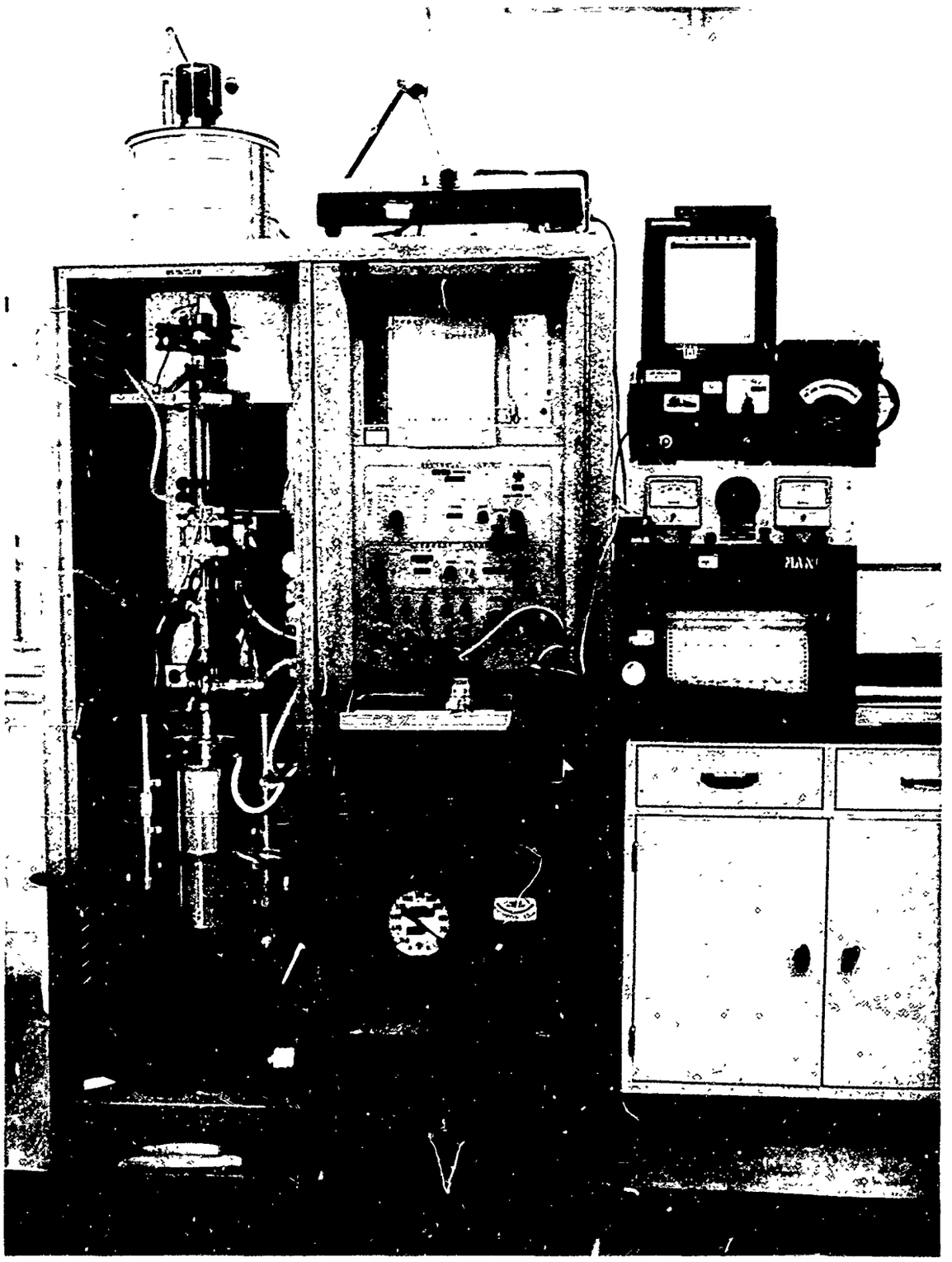

Figure 2. General View of the Recording Thermobalance After Modification

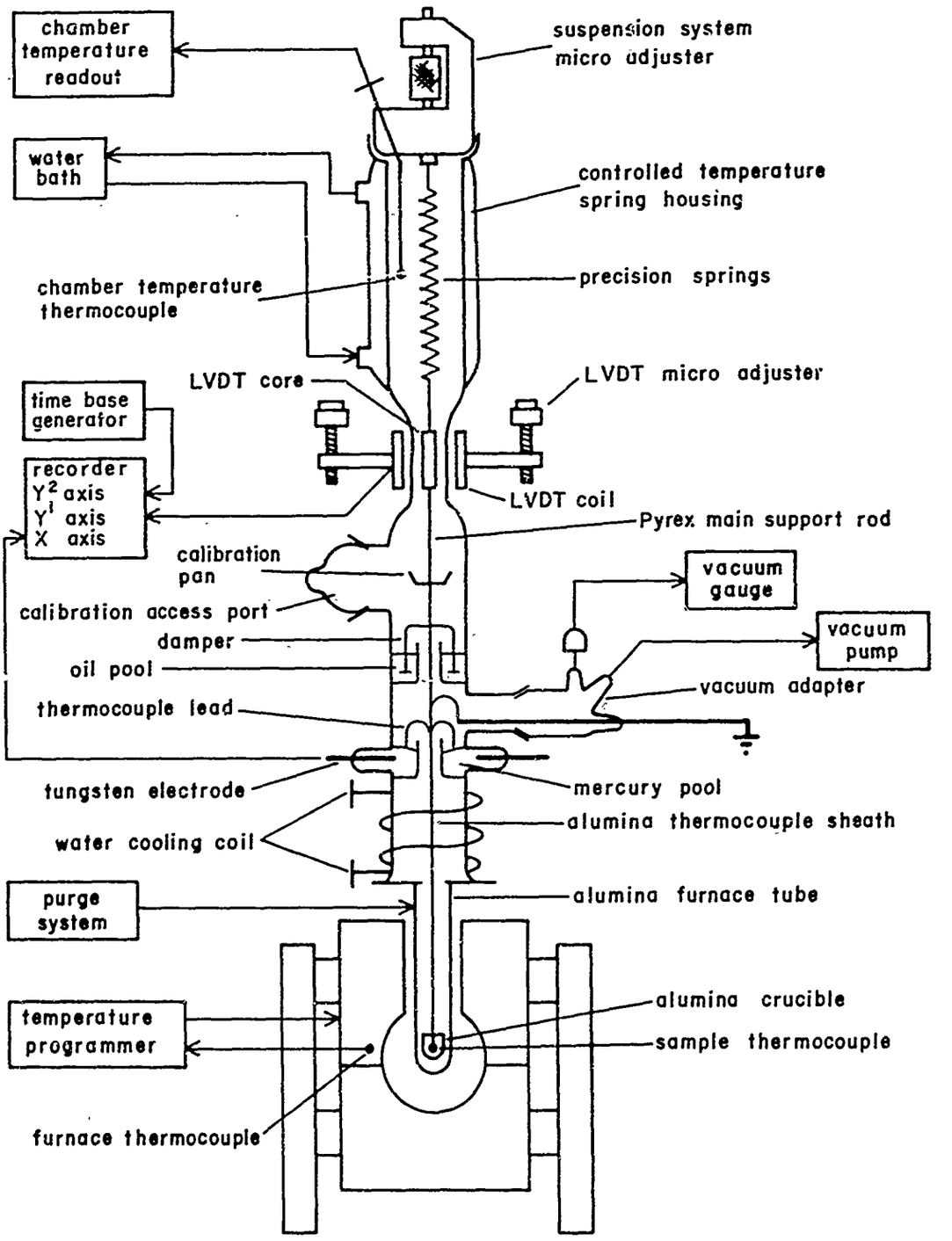

Figure 3. Recording Thermobalance Functional Diagram

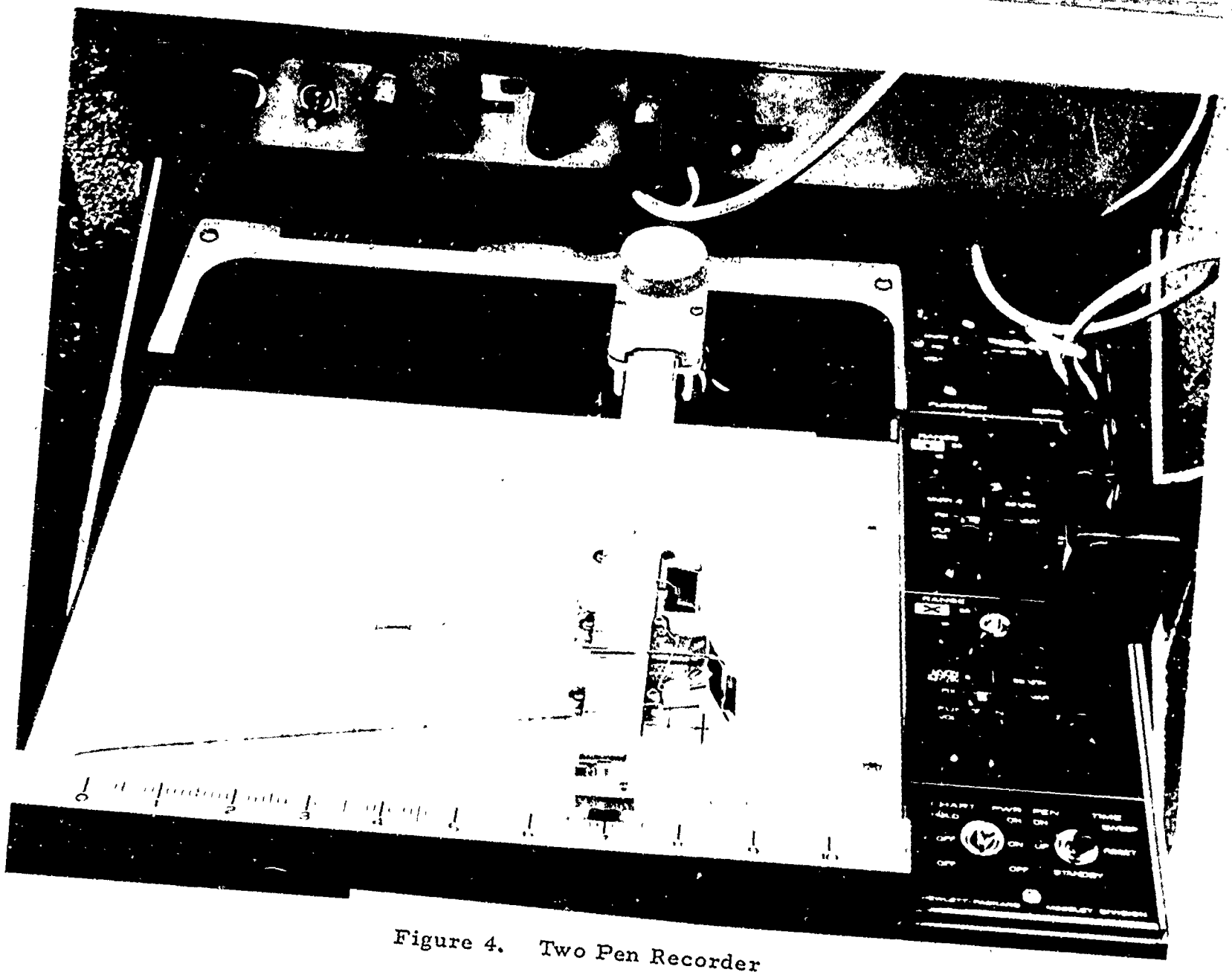

Figure 4. Two Pen Recorder

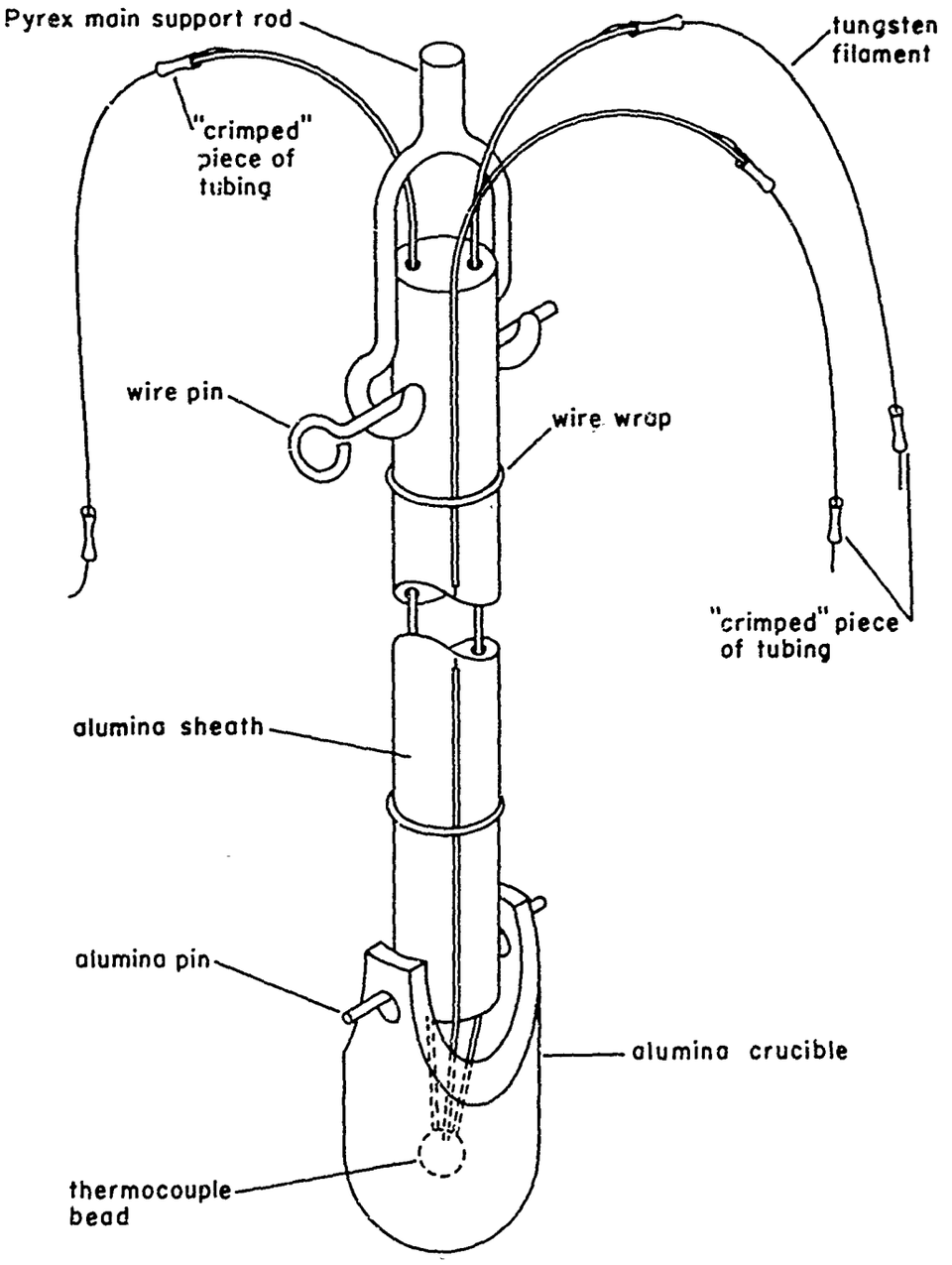

Figure 5. Thermocouple/Crucible Assembly

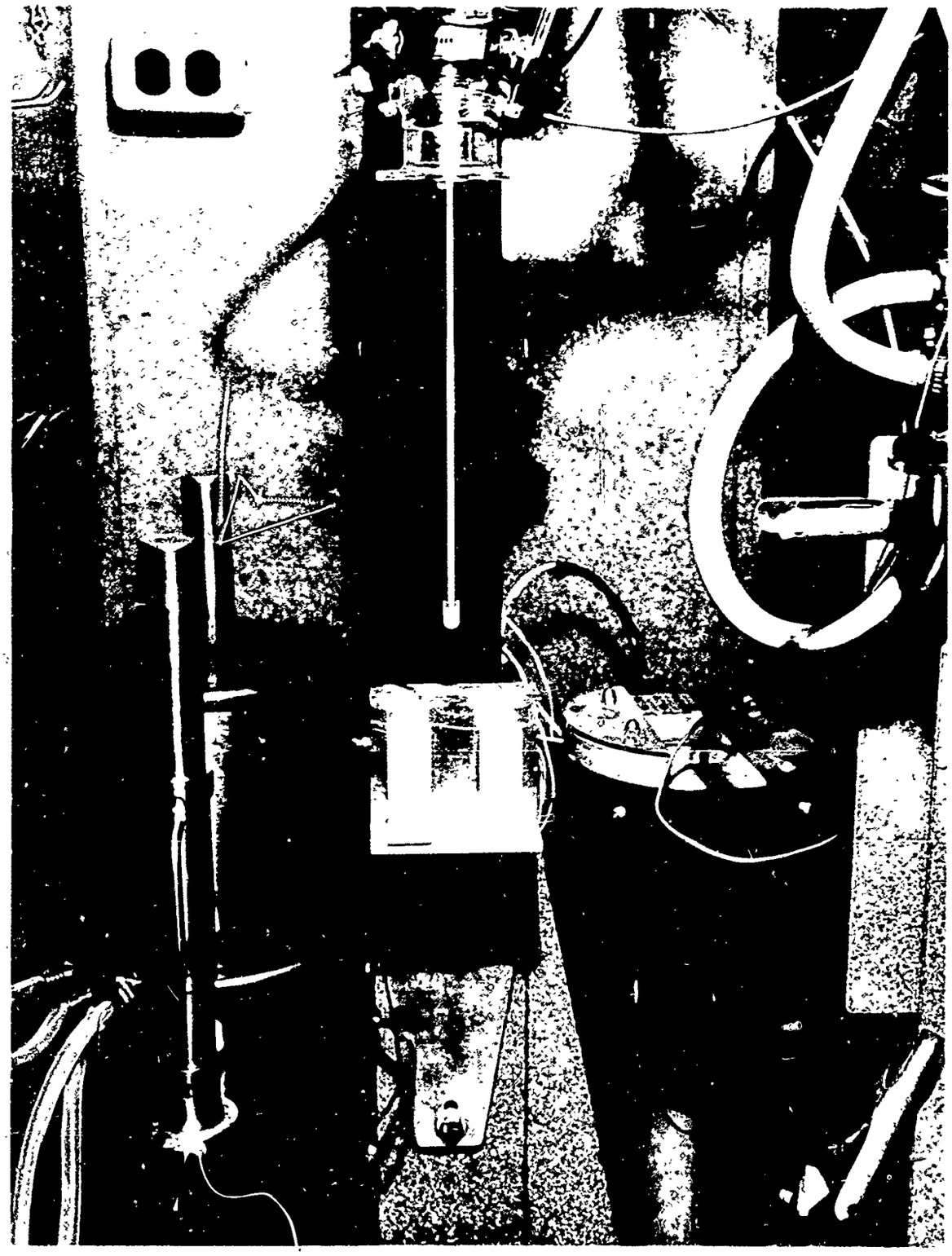

Figure 6. Thermocouple Sheath/Crucible Assembly and Crucible

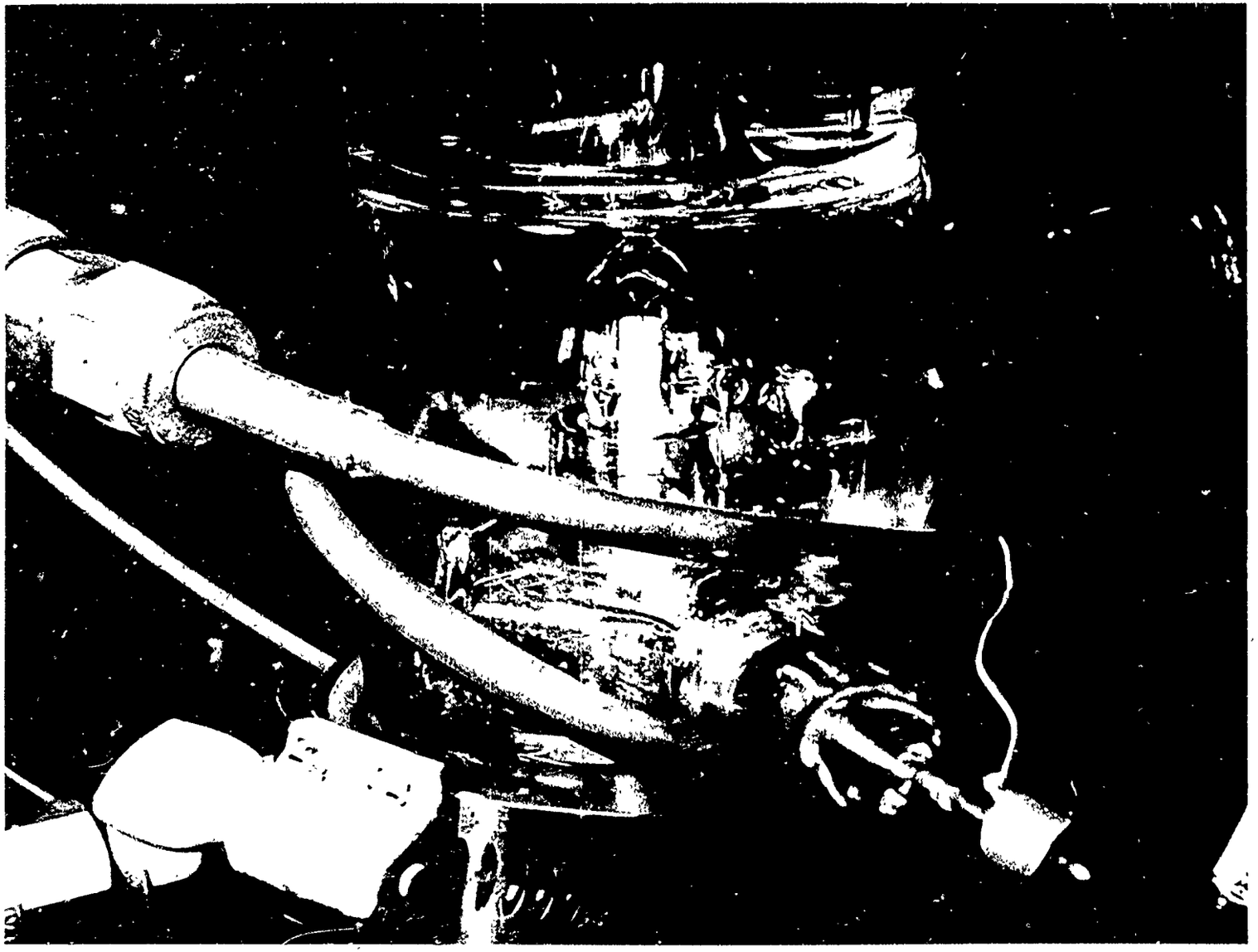

Figure 7. Mercury Pools, Thermocouple Leads, Tungsten Electrode and Stirrup

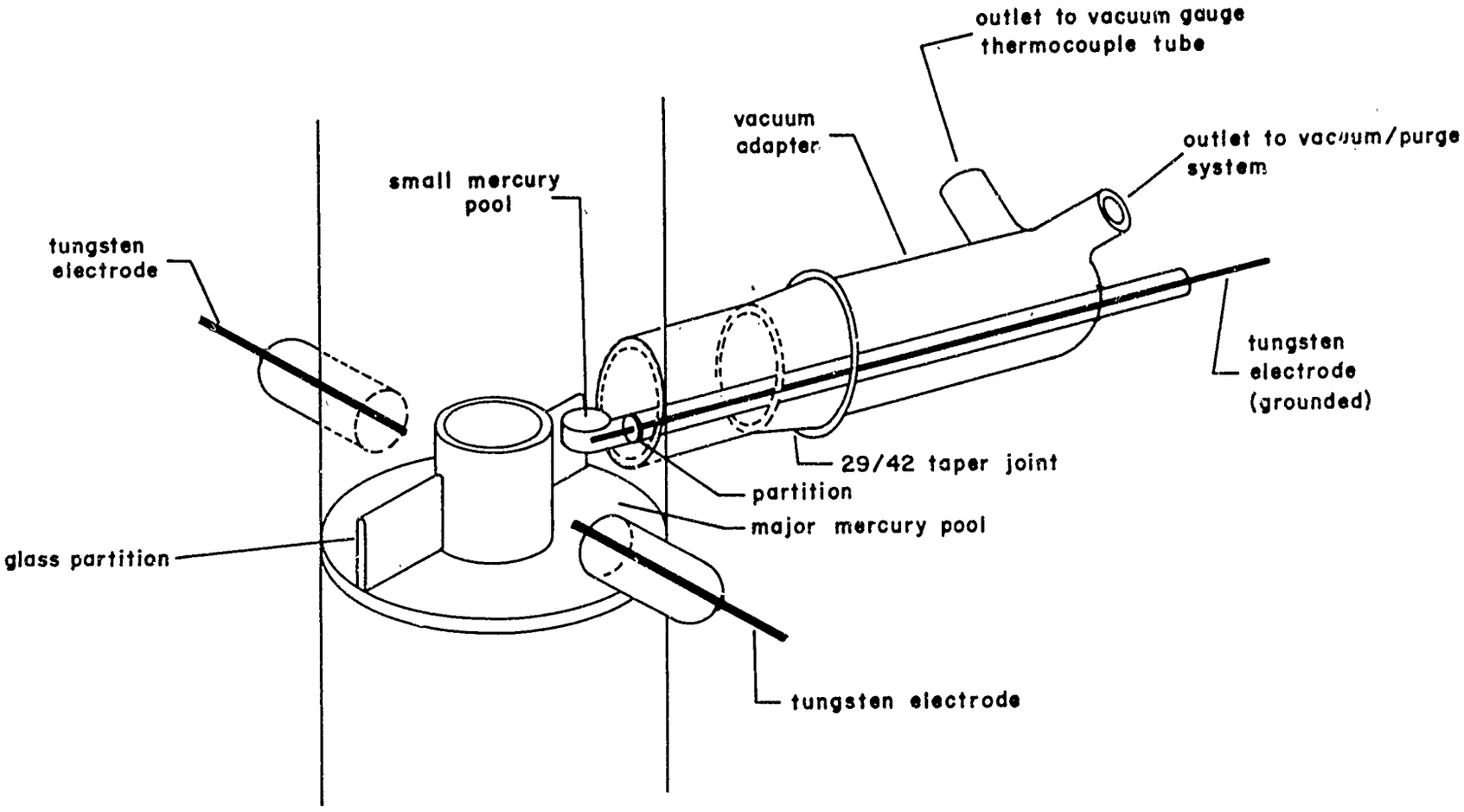

Figure 8. Mercury Pools and Vacuum Adapter

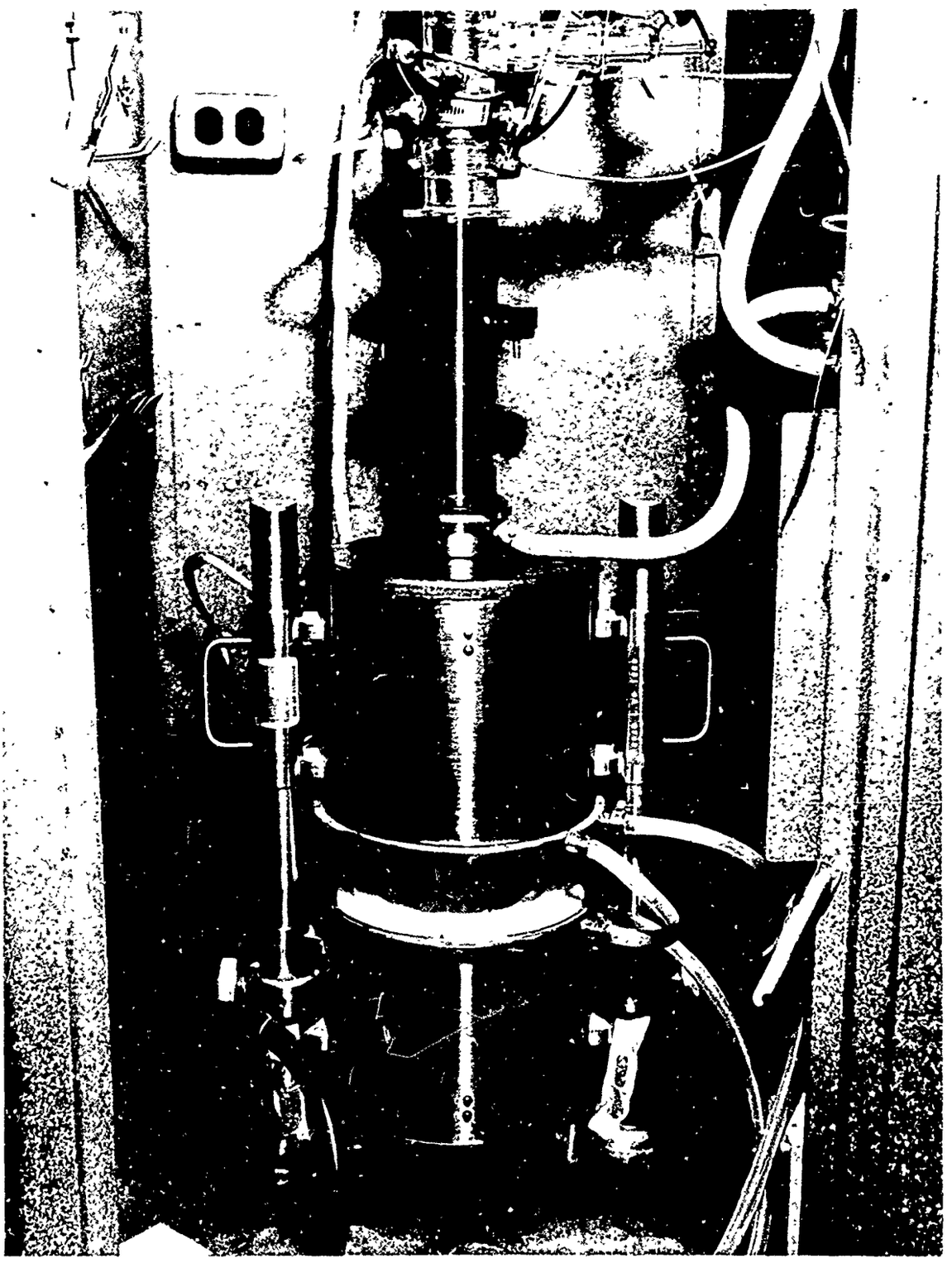

Figure 9. Tem-Pres Furnace, Open for Cooling

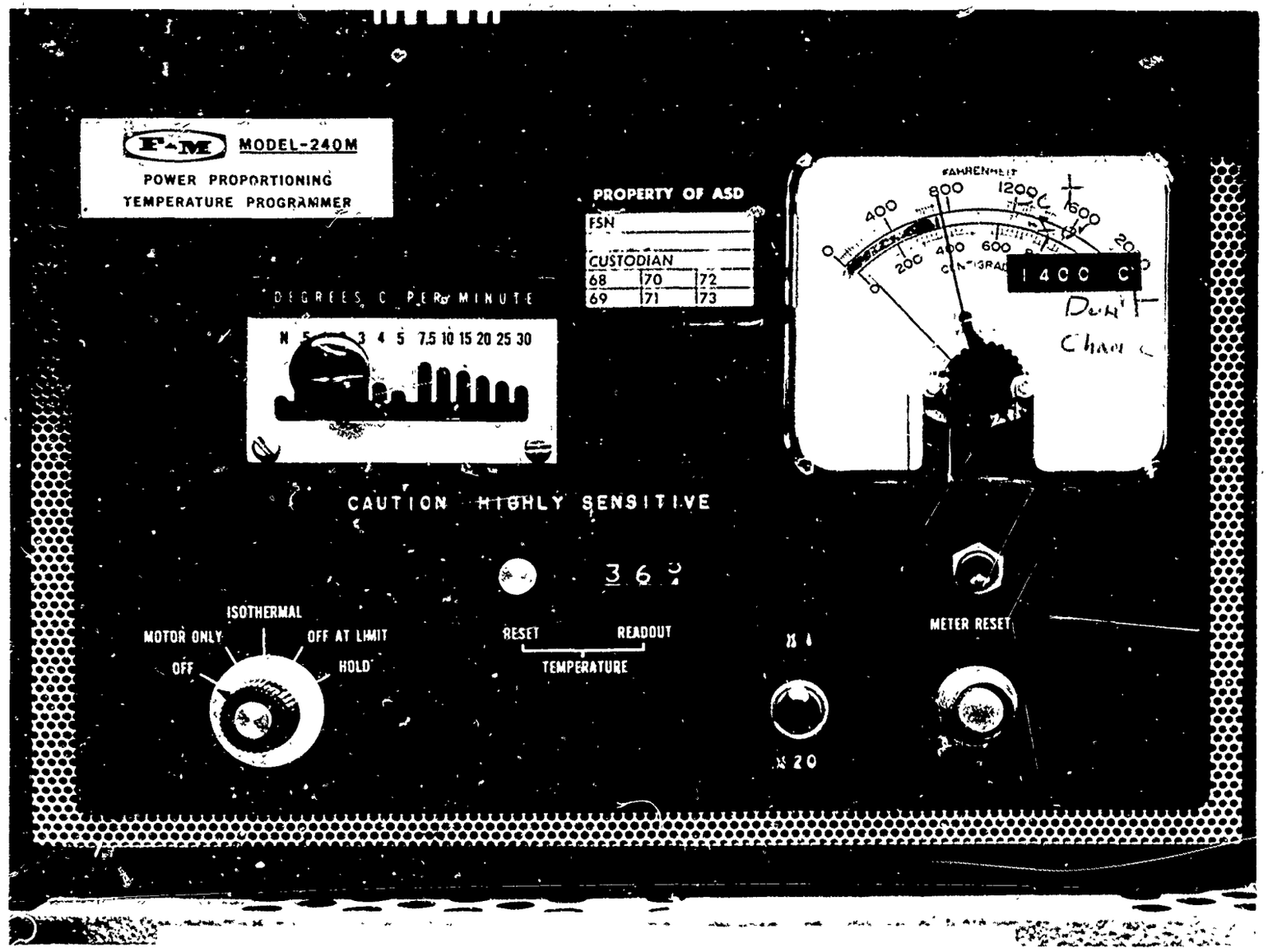

Figure 10. Power Proportioning Temperature Programmer

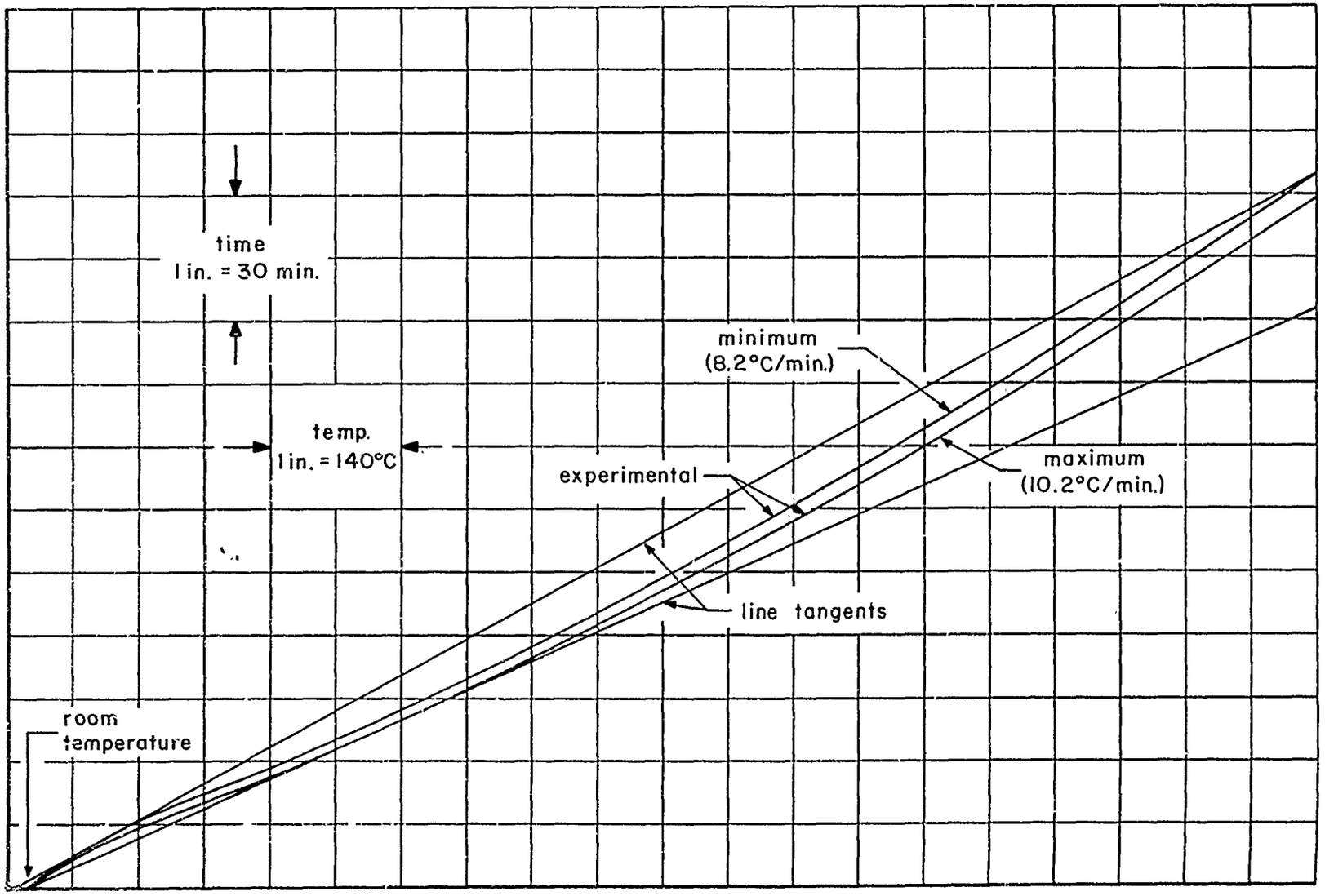

Figure 11. Heating Rate Linearity and Reproducibility

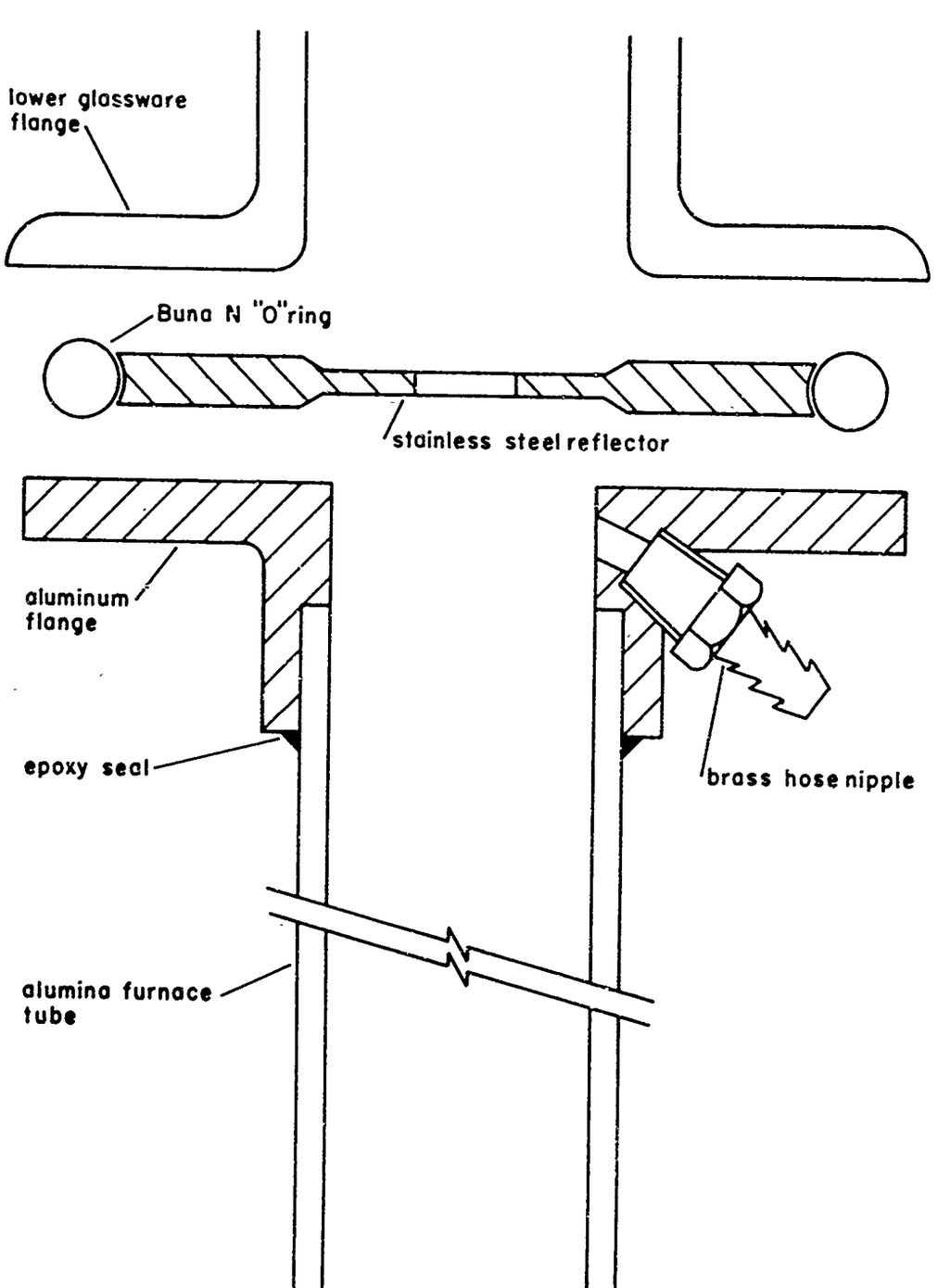

Figure 12. Furnace Tube and Vacuum Seal/Heat Reflector

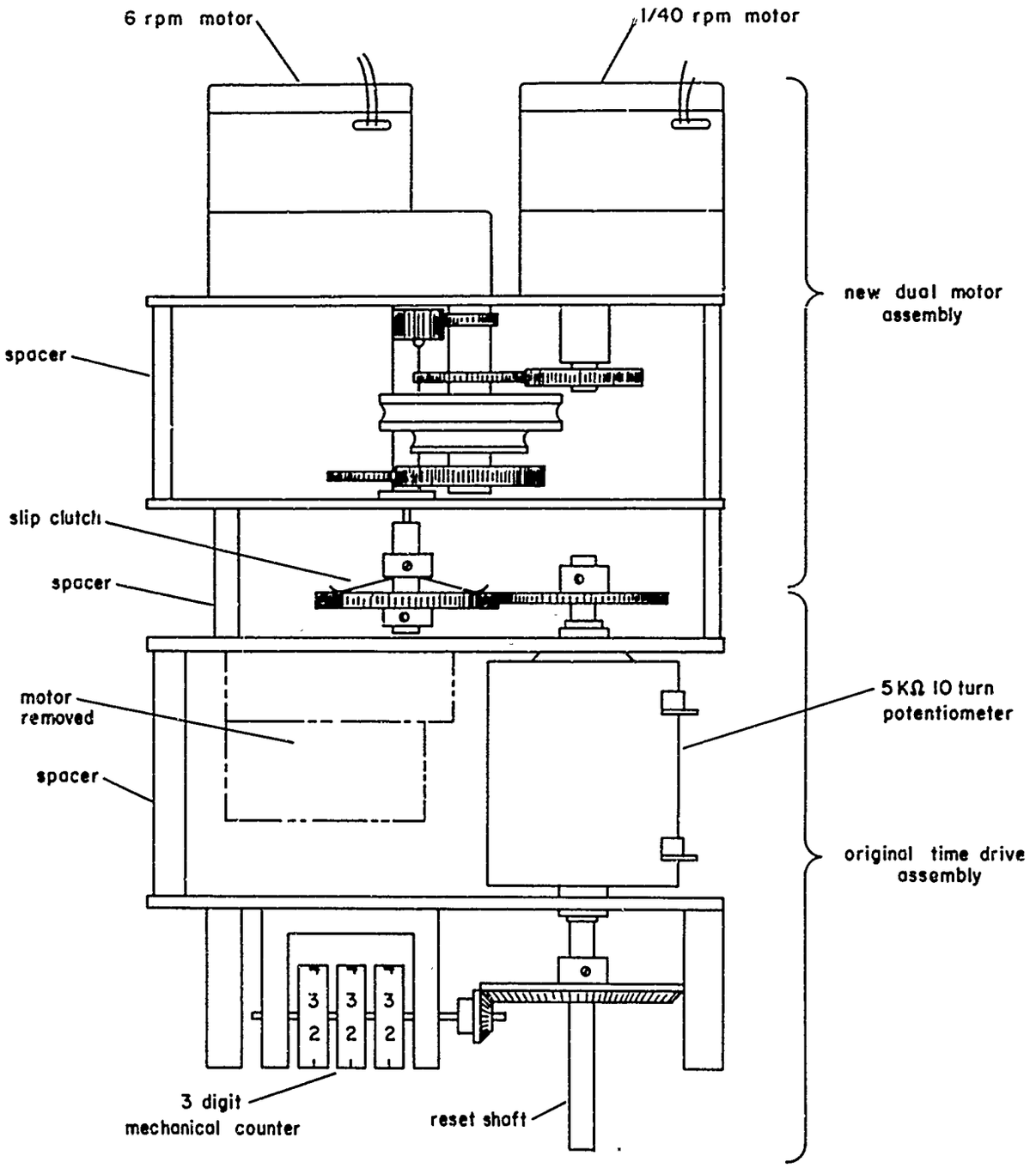

Figure 13. Time Drive Assembly

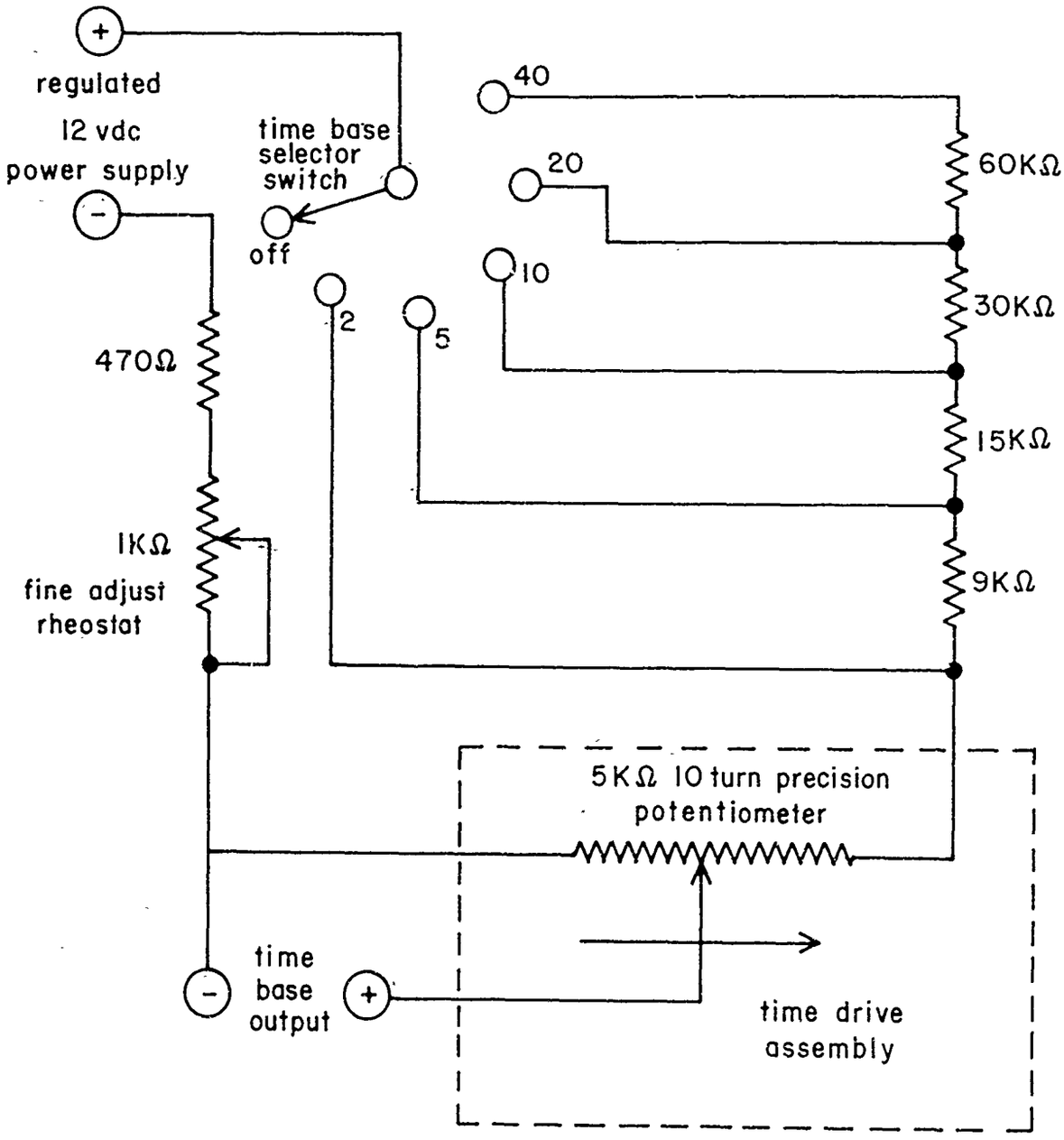

Figure 14. Voltage Divider

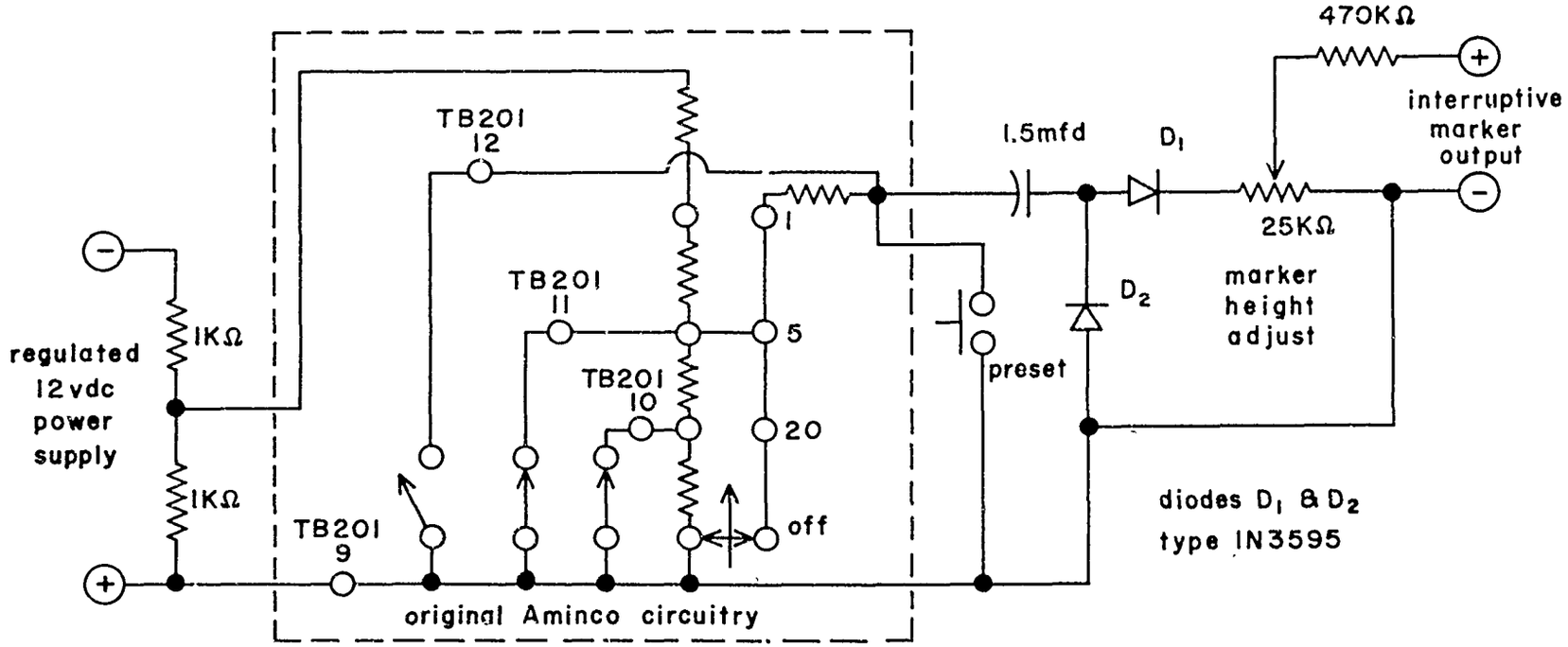

Figure 15. Interruptive Marker

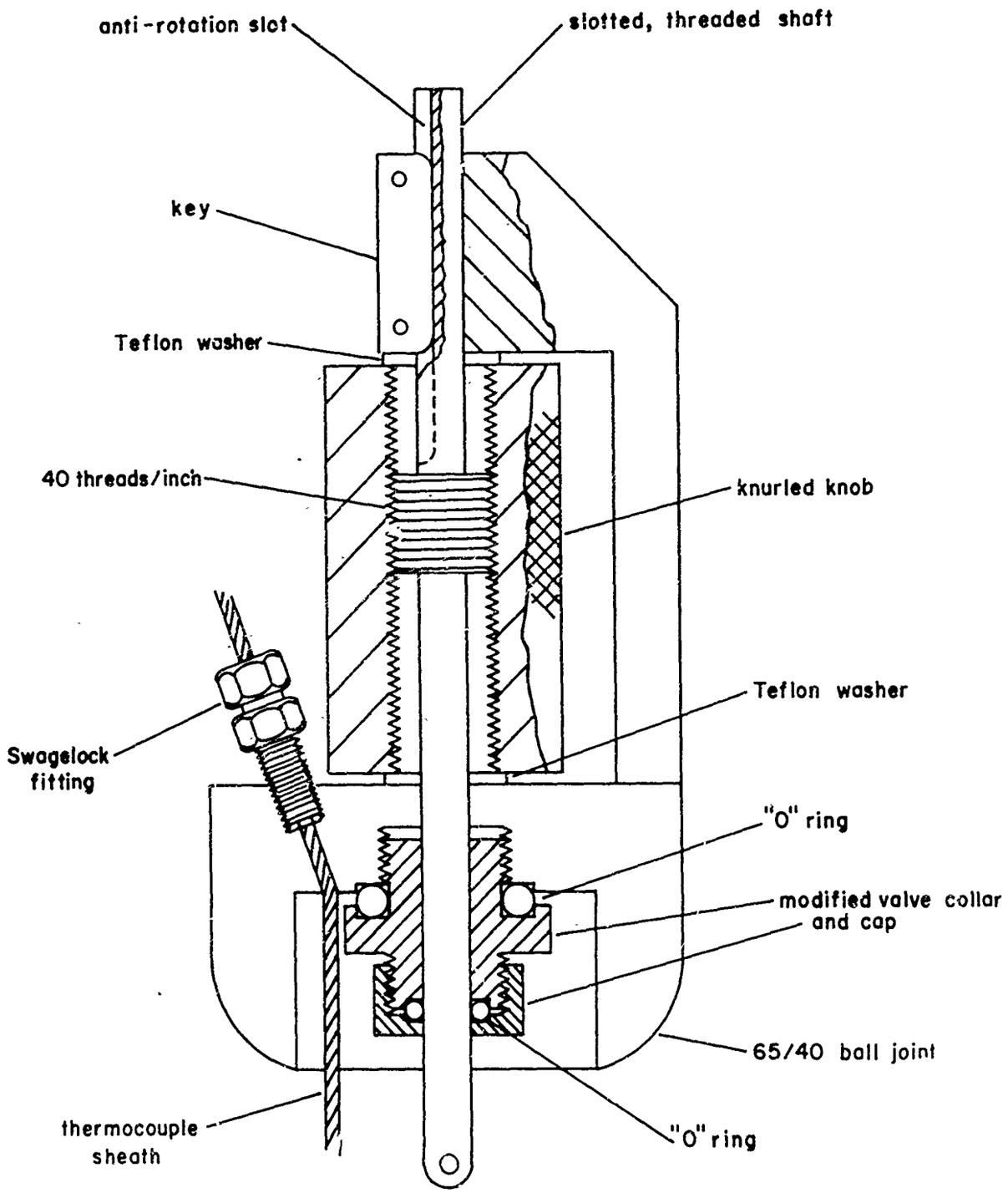

Figure 16. Suspension System Micro Adjuster

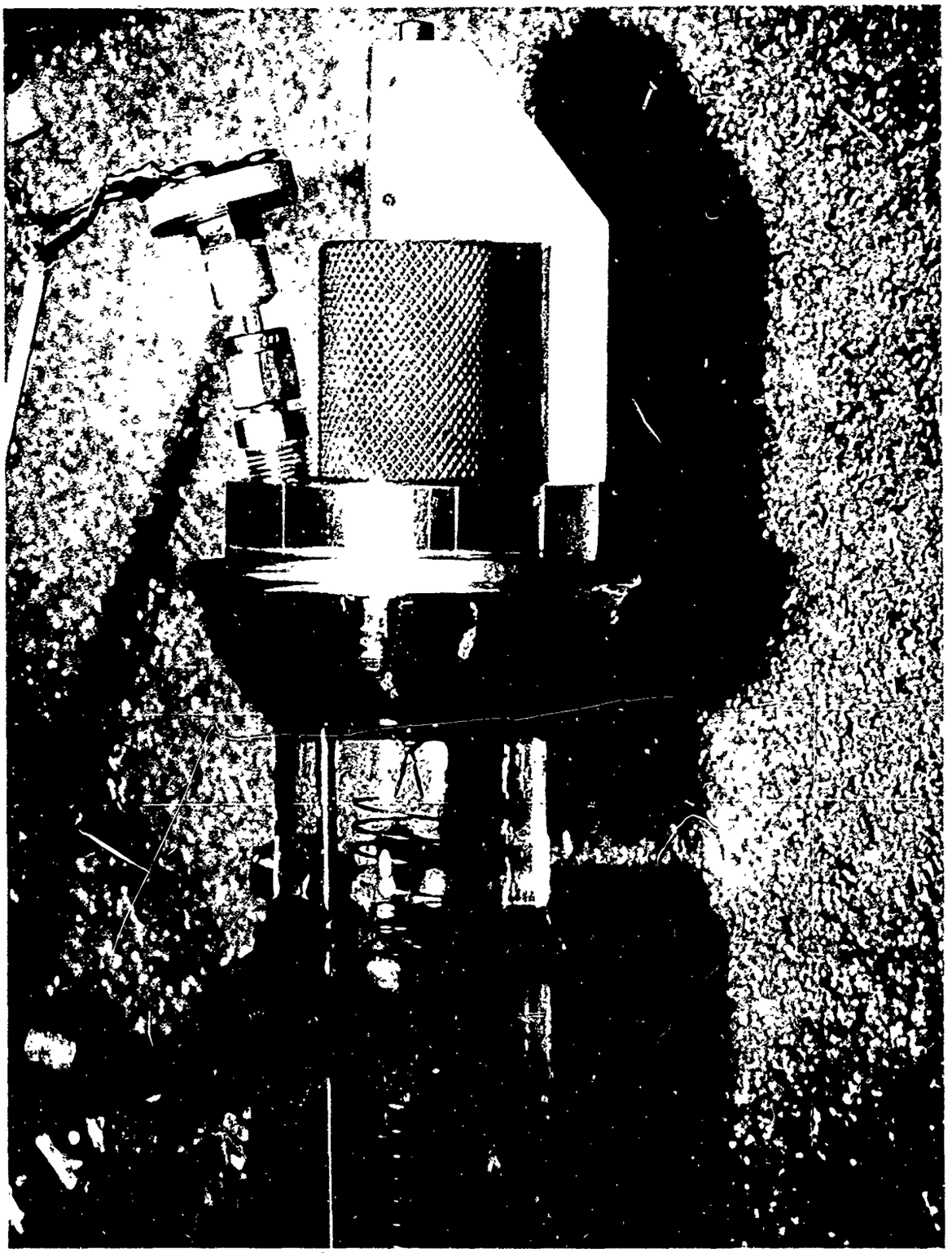

Figure 17. Suspension System Micro Adjuster in Place

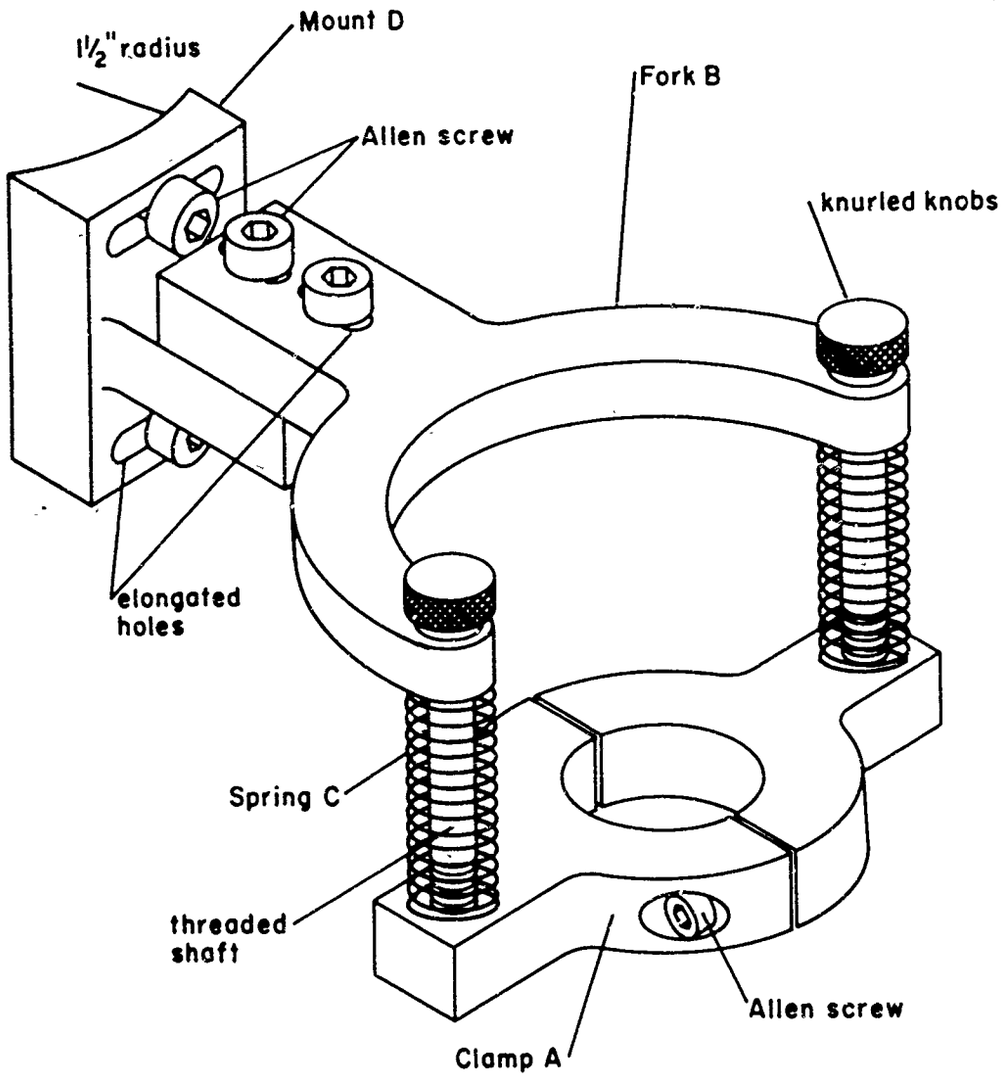

Figure 18. LVDT Micro Adjuster

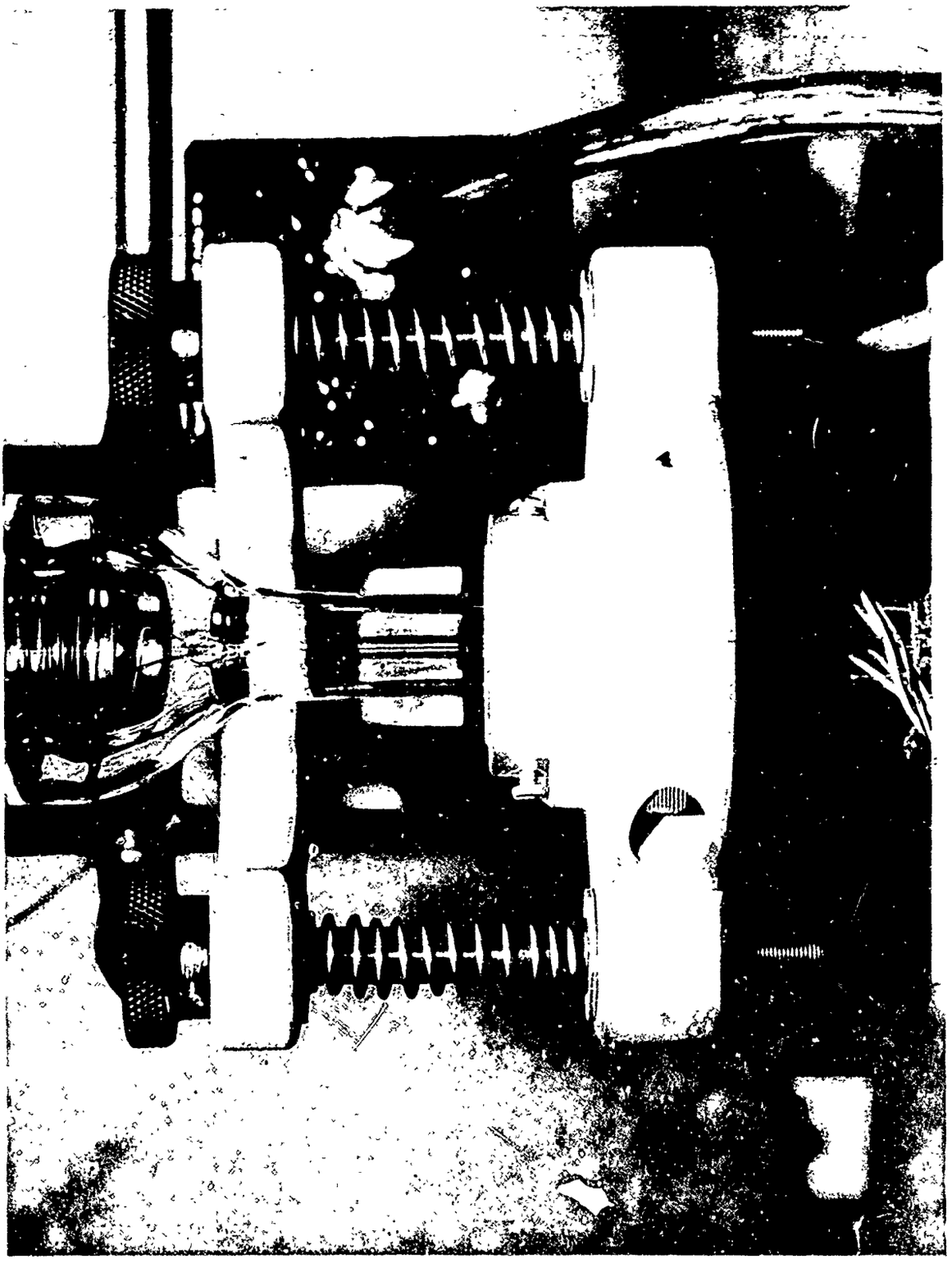

Figure 19. LVDT Micro Adjuster in Place

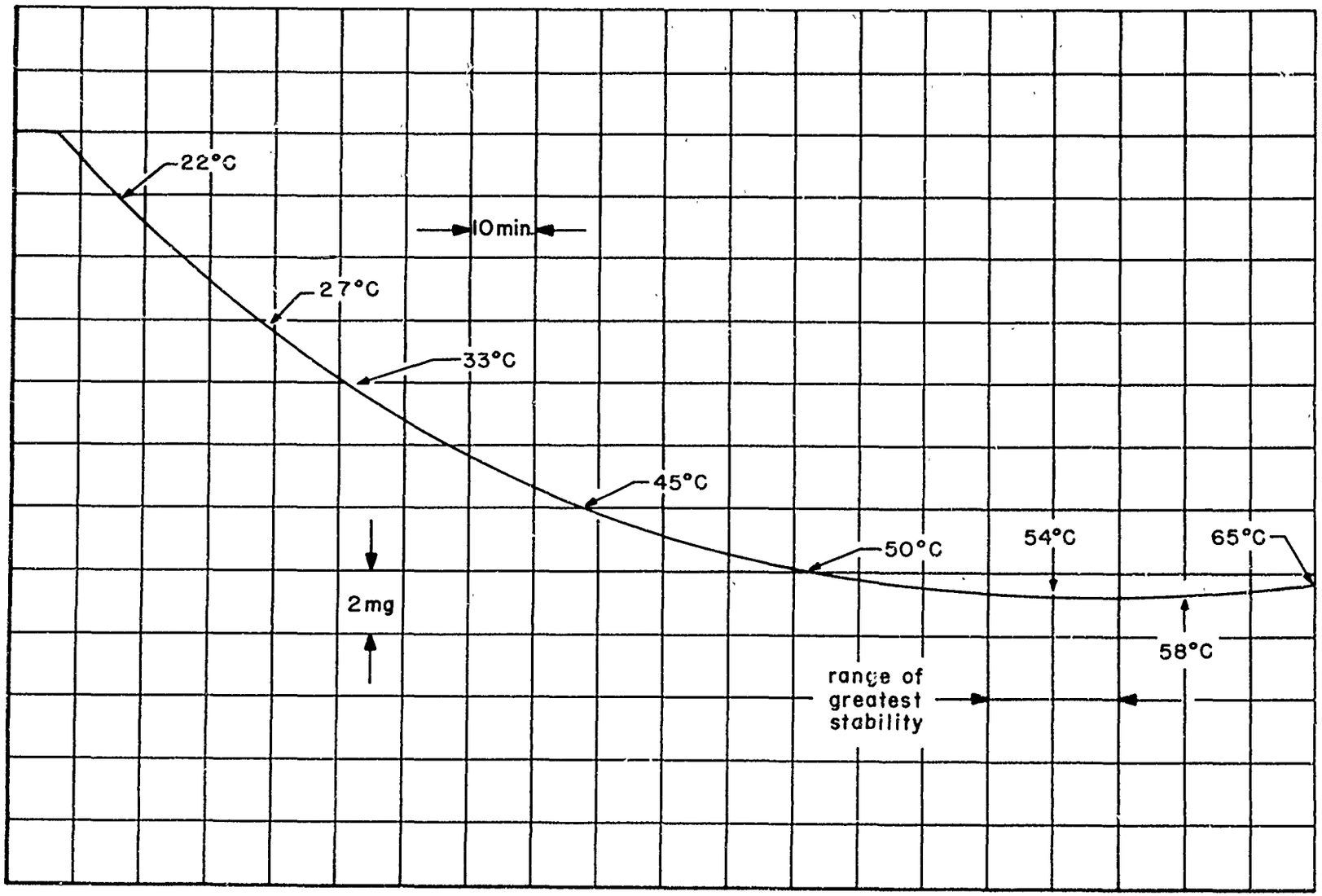

Figure 20. Temperature Effect on the Precision Springs

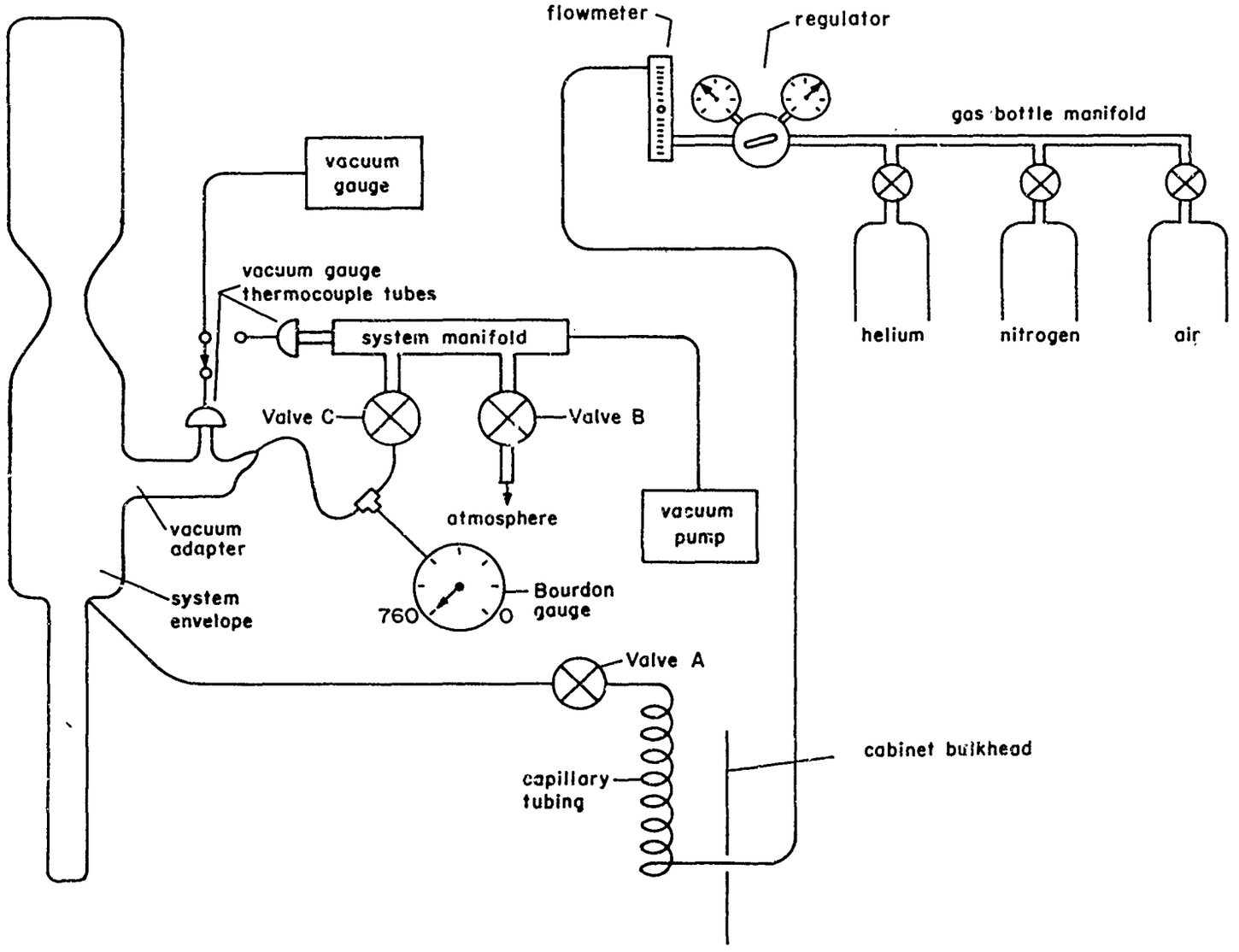

Figure 21. Vacuum/Purge System

Figure 22. Vacuum Pump and Vacuum/Purge Control Panel

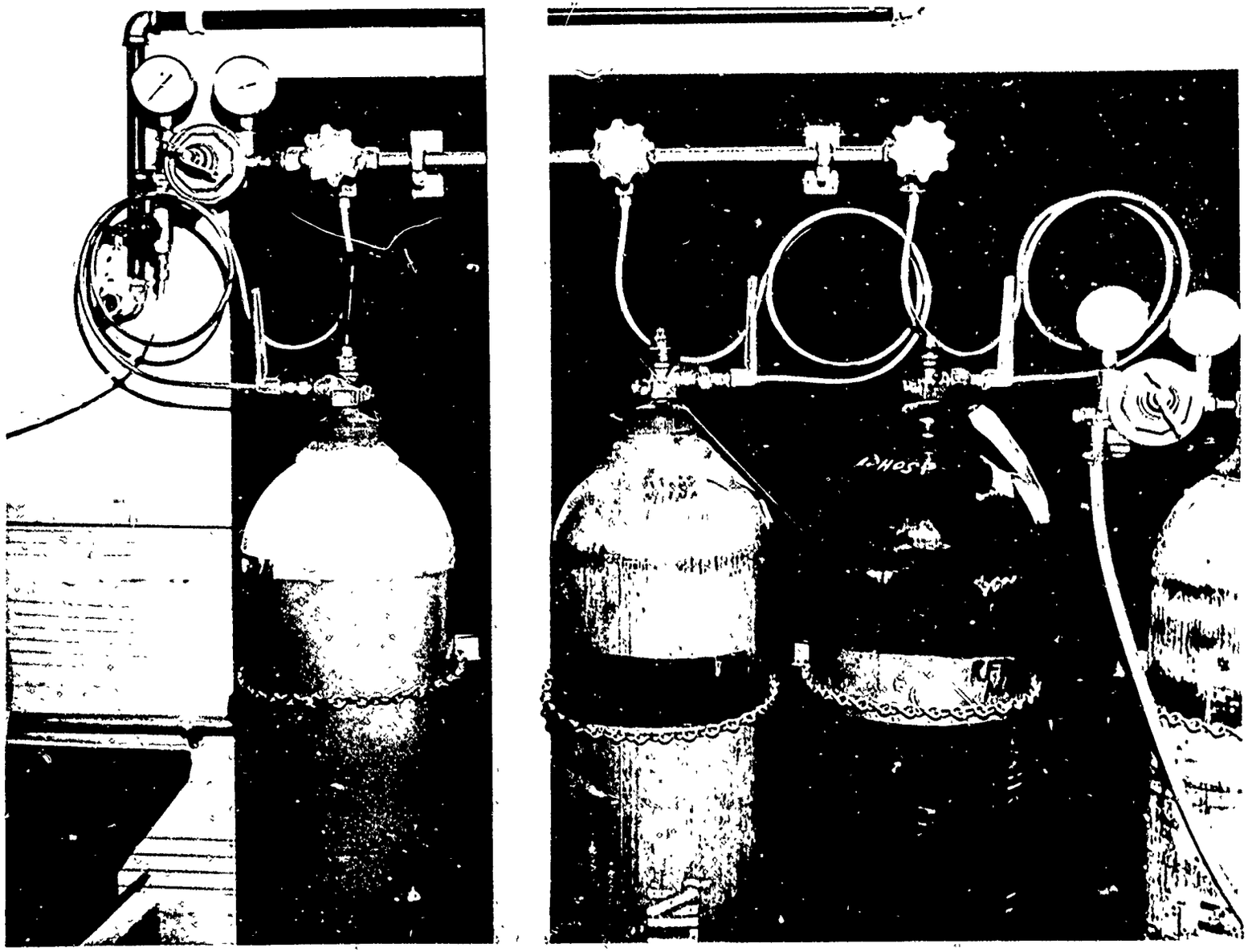

Figure 23. Gas Bottle Manifold

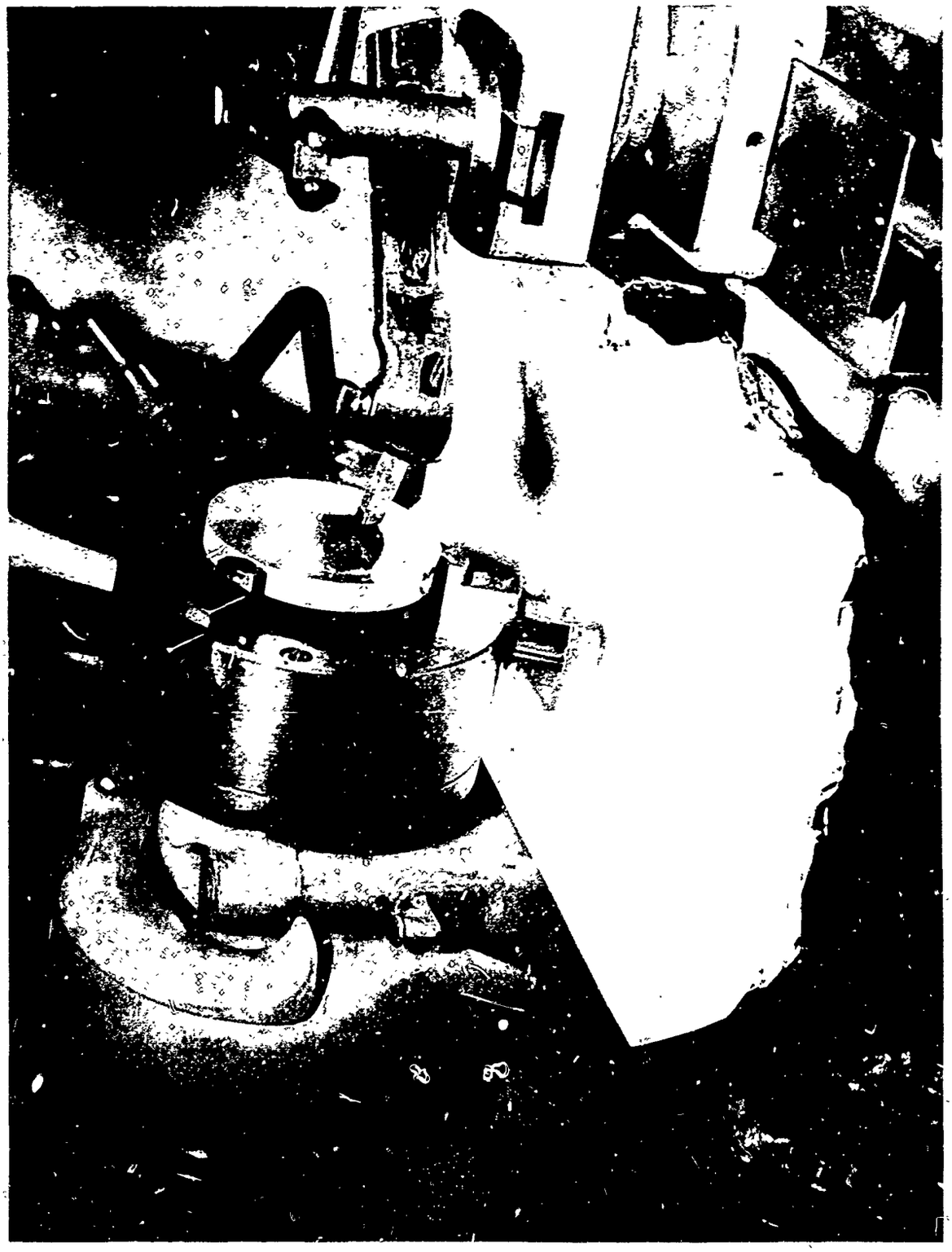

Figure 24. Sample Material Being Shaved in Lathe

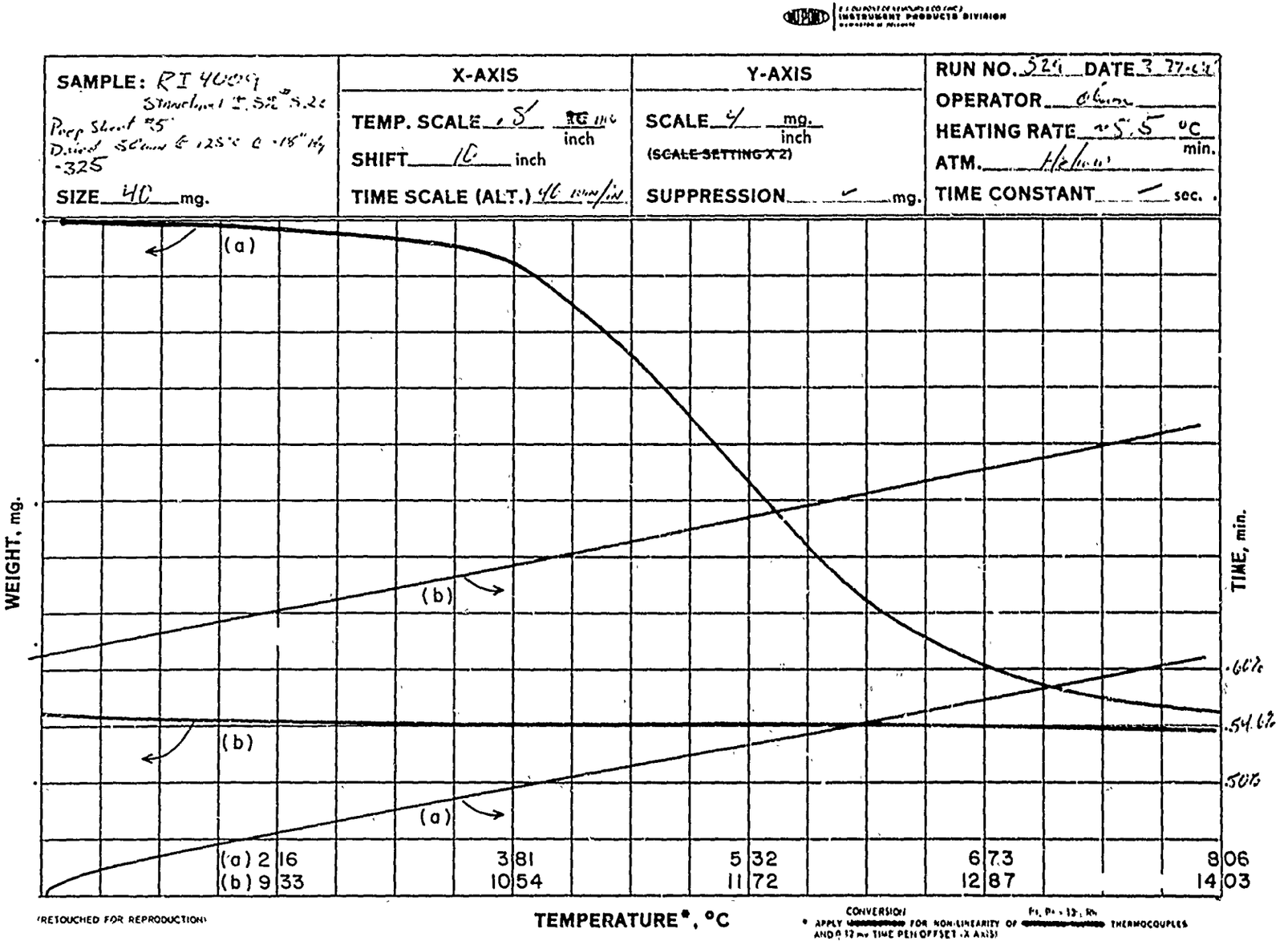

Figure 25. Thermogram of Phenolic Resin (RI 4009)

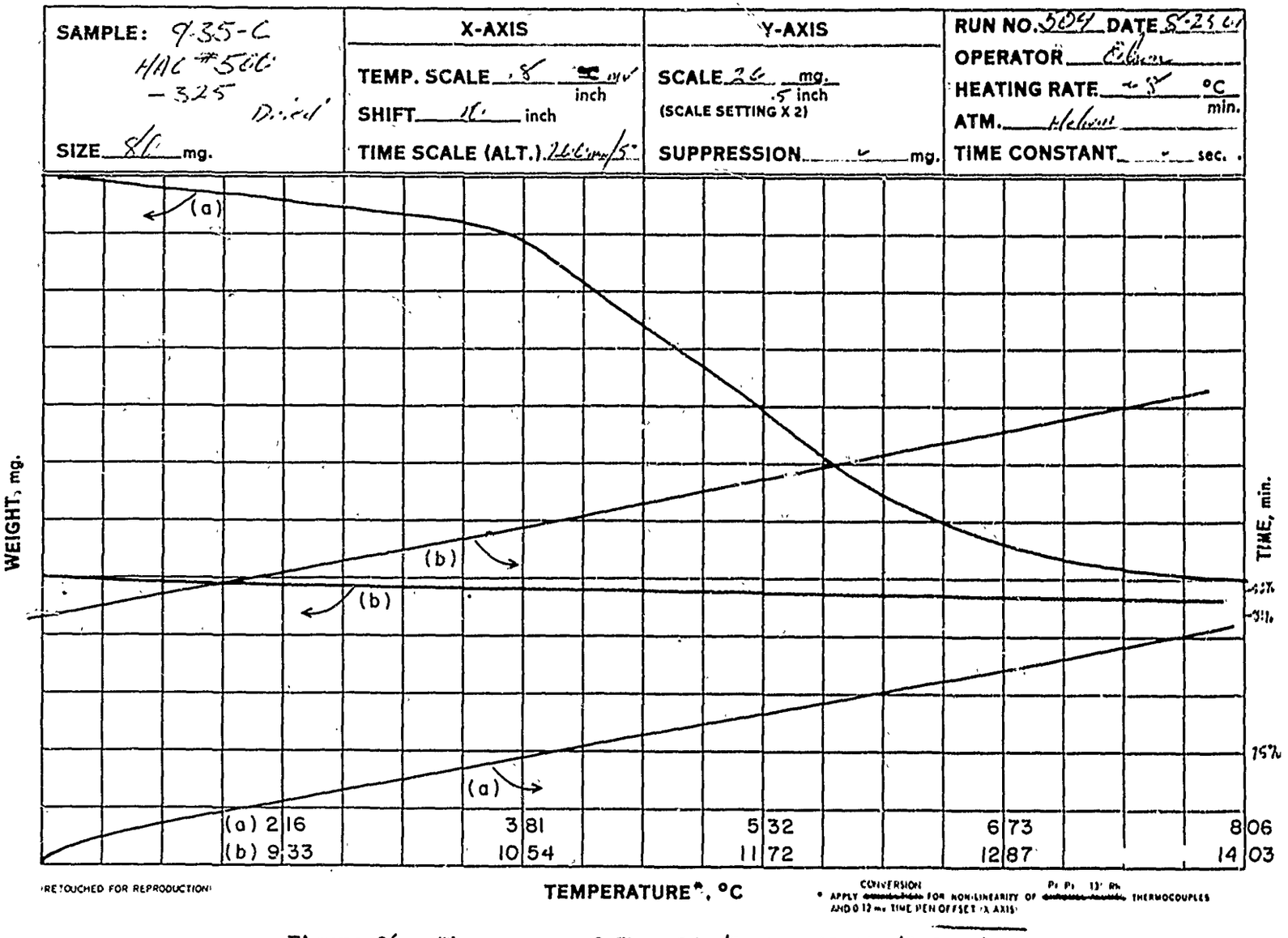

Figure 26. Thermogram of Phenolic/Carbon Cloth (9-35-C)

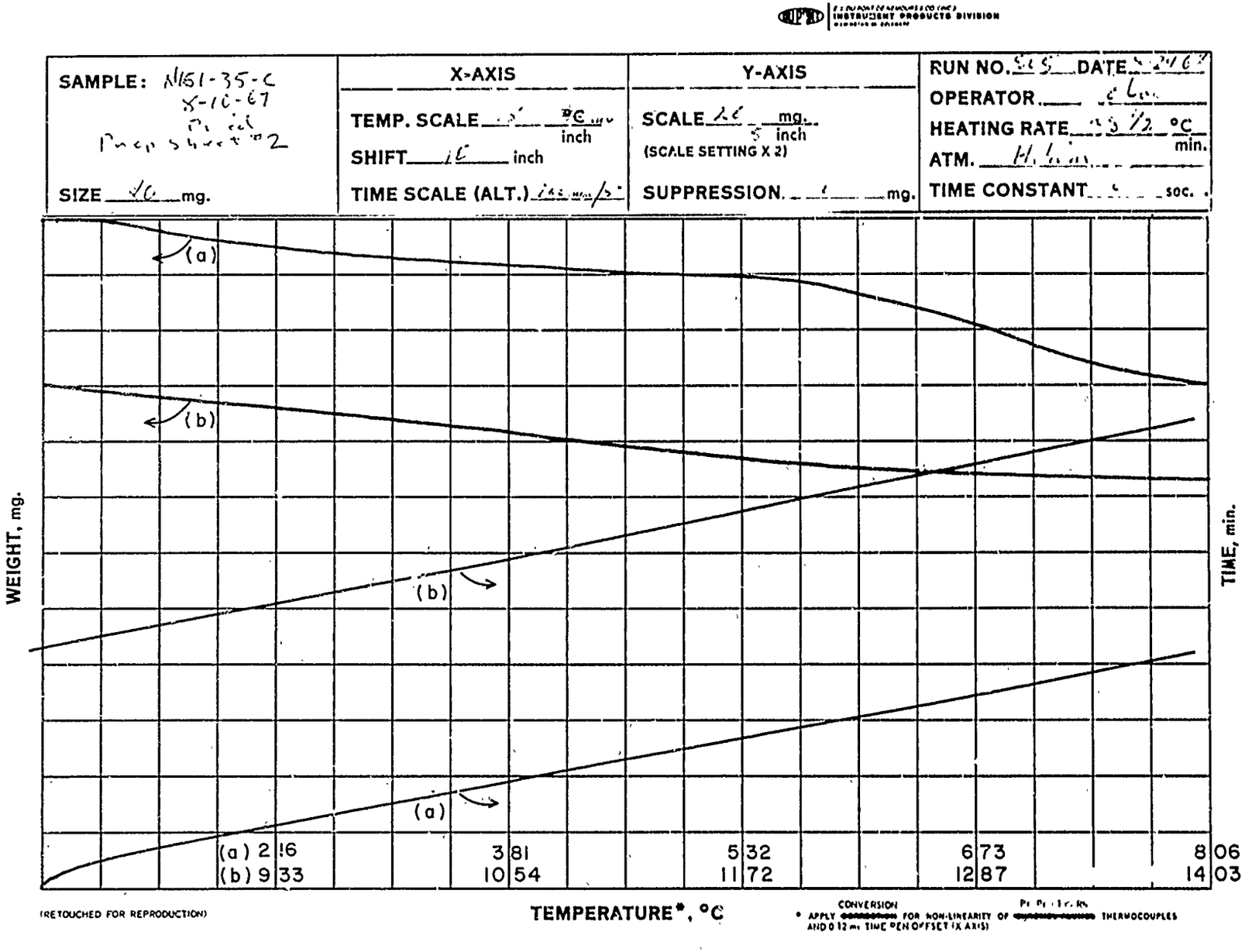

Figure 27. Thermogram of Polybenzimidazole/Carbon Cloth (N151-35-C)

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13. ABSTRACT This report describes a means of modifying a recording thermobalance for routine constant heating rate thermogravimetry to 1400°C with an optional temperature capability of 1750°C. During operation, a sample is suspended beneath a linear variable differential transformer (LVDT) system by precision springs. Any sample weight change varies the relative position of the springs resulting in a proportional voltage being generated by the LVDT system. The conditioned voltage is then plotted on the Y^1 axis of an X, Y^1, Y^2 recorder. Temperature is recorded on the X carriage axis via a platinum, platinum+13% rhodium, platinum (ground) thermocouple placed into the sample powder. A synchronous time base generator supplies a time readout for the Y^2 axis. The constant heating rate is closely regulated by a spherical furnace/SCR controller/time base recorder system. The atmosphere of the suspension chamber is air-freed and helium-filled by room temperature evacuation/purge cycles before the run. A run typically involves a -325 sieve 80 mgm dried powder sample to 1400°C at 8.5°C/min in helium purging flow. Recorder ranges are typically 0-25 mg/5 inch, 0-1400°C/20 inch, and 0-200 min/5 inch.

In this thermobalance, the thermocouple wires are insulated by an alumina sheath which also serves as the crucible holder. Tungsten wire/mercury pool/tungsten rod electrical contacts result in minimum spring suspension effect. The thermocouple signal is filtered and grounded to shield against electromagnetic furnace noise normally found at high temperatures.

(Continued on attached sheet . . .)

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ABSTRACT (continued)

Typical thermograms are given for phenolic resin and polybenzimidazole/carbon cloth laminate samples. The latter material, which is of interest for hyperthermal ablative use, underwent pyrolysis beyond the typical analysis limit of 1000°C and approached a constant weight at 1400°C. The poor grinding qualities of reinforced plastics required a unique lathe machining method to produce a fine powder sample.

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	ROLE	WT	ROLE	WT	ROLE	WT
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